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ERRATA.

The second paragraph which appears on page 124 is misplaced ; it should be read in connection with the table on page 120, to which it refers.

Page 282, lines 17, 19, and 20. *for* "per cents." *read* "parts per thousand."

Page 326, lines 6 and 9 from bottom, *for* "sulphur chloride" *read* "carbon disulphide."

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THE ANALYST.

JANUARY, 1897.

PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS.

THE monthly meeting of the Society was held on Wednesday evening, December 2, 1896, at the Chemical Society's Rooms, Burlington House, the President occupying the chair.

The minutes of the previous meeting were read and confirmed.

Mr. David James Morgan, B.A., was elected a Member of the Society, and Messrs. Percy T. Adams, Louis Charles Deverell, and Percy S. Marshall were elected Associates.

Mr. Frank R. Stephens, analyst to Messrs. Idris and Co., was proposed for election as a Member.

Mr. BEVAN (Hon. Sec.) read the following list of officers for 1897, as nominated by the existing Council:

President: Bernard Dyer, D.Sc.

Vice-Presidents who have filled the office of President: M. A. Adams, F.R.C.S., A. H. Allen, Sir Chas. A. Cameron, M.D., F.R.C.S., A. Dupré, Ph.D., F.R.S., Otto Hehner, Alfred Hill, M.D., F.R.S.E., J. Muter, M.A., Ph.D., F.R.S.E., Thos. Stevenson, M.D., F.R.C.P.

Vice-Presidents who have not filled the office of President: A. P. Aitken, D.Sc., F.R.S.E., W. W. Fisher, M.A., John Pattinson.

Hon. Treasurer: E. W. Voelcker.

Hon. Secretaries: E. J. Bevan, Charles E. Cassal.

Other Members of Council: R. Bodmer, A. Wynter Blyth, M.R.C.S., A. C. Chapman, S. Rideal, D.Sc., J. E. Stead, J. A. Voelcker, M.A., B.Sc., Ph.D.

The names of those members of the Council who do not retire this year are Leonard Archbutt, Bertram Blount, E. Russell Budden, W. J. Dibdin, Sidney Harvey, and Alfred Smetham.

The following papers were read: "Note on Formalin," by Ronald Orchard; "On the Separation and Estimation of Formic Aldehyde," by Norman Leonard, B.Sc., and Harry M. Smith; "Notes on Prussian Blue," by Frank H. Leeds; "The Estimation of Borax and Boracic Acid in Milk," by Frank P. Perkins; "Note on Copper in Oysters," by W. F. Lowe; "The Statement of Analytical Results,' by J. F. Liverseege.

A FURTHER NOTE ON LEAD IN CANADIAN CHEESE.

By F. Wallis Stoddart, F.I.C., F.C.S.

(Read at the Meeting, November 4, 1896.)

On page 208 of the current volume of the Analyst is a report of a short communication I made to the Society on cheese containing lead in what I believed to be largely the metallic condition. The evidence on this point, however, was not of the most conclusive character, as was pointed out by several contributors to the discussion that followed.

The physical characters of the material seemed to limit the possibilities to the metal more or less superficially corroded, the sulphide, and to certain salts such as the carbonate blackened by sulphide resulting from decomposition of the cheese. The difficulty in deciding arose, of course, not so much from the want of analytical methods, as from the impossibility of freeing the matter from adherent particles of cheese. It should be remembered that the total lead amounted only to 1¼ grains per pound of cheese, that most of this was so distributed over the surface of the curds as to be inseparable by scraping, and that any attempt at destruction of the adherent matter defeated the object of the experiment by attacking the pigment.

My reasons for arriving at the conclusion stated were the solubility relations to acids, particularly to dilute nitric acid in the cold, the purity of the oxide obtained on ignition, and the entire failure to detect sulphur in any shape; to these may be added the readiness with which the substance was attacked by caustic alkali in attempts to purify it.

As regards the detection of sulphide, it was suggested by Mr. Allen that a good method consisted in treating with hydrochloric acid in presence of zinc, when sulphuretted hydrogen is readily liberated. This reaction has been applied without demonstrating the presence of sulphide. It seemed, therefore, very desirable to obtain positive evidence as to the condition of the lead, and further consideration has suggested a method which apparently yields decisive results.

It is well known that lead ranks amongst the more electro-positive metals, and that many others, especially silver and copper, are readily deposited upon it from slightly acid solutions. A stick of lead immersed in a slightly acid solution of copper is speedily covered with a closely adherent film of characteristic colour. If this film is scraped off with as little lead as possible, and the metals precipitated from solution in acetic acid with a ferrocyanide, the lead ferrocyanide formed is tinged more or less deeply with the red copper ferrocyanide. This forms an exceedingly delicate test for copper, the least shade of red being very perceptible. Under parallel circumstances, silver is deposited as a brownish black pulverulent coating rather easily detached by rubbing.

It appeared to me that these reactions would be applicable to the problem in hand. By working over ten or twelve pounds of cheese, and selecting the best bits, about half a gramme of material rich in the black pigment was obtained. This was rubbed down in alcohol, and, after decantation of the latter, thoroughly exhausted with ether; it was then washed successively with alcohol and water. After decanting

as closely as possible, the moist residue, consisting now of the pigment with perhaps five times its bulk of casein, was divided into two parts, and treated with silver nitrate and copper sulphate respectively. In a few minutes a distinct change of colour was observable in the silver experiment, the mass taking a brownish shade. Both tubes were left for thirty minutes, then washed with hot water until the washings gave no reactions for metals; the residues were then treated with warm dilute nitric acid, when the filtrates gave abundant reactions for silver and copper respectively. The precipitate of silver chloride so obtained weighed 9 milligrammes, representing about 6 milligrammes of metallic lead. The reaction appeared less complete in the case of the copper.

Now, a possible objection—the only one, as far as I can see—to this experiment is based upon the known affinity of proteids for the heavy metals, notably for copper; and it may be suggested that the casein remaining adherent to the pigment may so far fix either copper or silver as not to yield it to water, though it may subsequently do so to nitric acid. Blank experiments, therefore, were carried out side by side with that described above, using a considerably greater quantity of (*a*) sound portions of the cheese under examination, and (*b*) cheese from an entirely different source. Of these blank experiments, those with silver yielded in the final stage a distinct opalescence of chloride, showing that a trace of silver was rendered insoluble in water. No reaction was obtained with the copper under the same conditions, but a more rigid examination showed that a minute quantity of copper was fixed. The amount, however, in both cases was so minute in comparison with that obtained in presence of the black pigment, as to be negligible for the special purpose in view.

I think, therefore, I am justified in concluding that the black pigment consists essentially of metallic lead, with possibly some superficial sulphide.

As to the origin of this impurity, no light has up to the present been obtained. The most reasonable suggestion seems to be that it is introduced accidentally by friction upon some leaden surface. This explanation would be consistent with the fact that out of a consignment of two hundred cheeses about one hundred and fifty were affected in varying degrees.

An interesting point is that for many months mice refused to eat this cheese, though they repeatedly disposed of the greasy paper in which it was wrapped; and only very occasionally, and to a very limited extent, was the cheese itself attacked. On the other hand, during the last few weeks it has become such a happy hunting-ground for "jumpers," the larvæ of a small black fly, as to necessitate its destruction.

Discussion.

Mr. Allen said it was no doubt a very curious thing that the lead should be present, and one began to speculate as to its origin. He had heard of cheese being wrapped in cloths impregnated with chromate of lead, and it occurred to him to ask if Mr. Stoddart had looked for chromium in any form. It was possible, in the case of the heavy metals, that they might be reduced from such a compound as a chromate to a metallic state by reaction with the organic constituents of the cheese. Personally he was not satisfied as to the condition in which the lead existed, but Mr. Stoddart had taken a great deal of trouble in the matter, and it was not easy to decide as to

the condition of a substance existing in such a finely-divided state and such small quantity in an article like cheese. Whatever its origin might be, however, there could be no doubt as to its existence, and it behoved public analysts to do all they could, in cases like this, to ascertain, if possible, how the impurity came to be present.

NOTE ON THE ESTIMATION OF FORMALDEHYDE.

By Ronald Orchard.

(Read at the Meeting, December 2, 1896.)

Owing to the rapidly increasing importance of formaldehyde, both commercially and as a laboratory reagent, a simple and exact method for determining the strength of its dilute solutions is much needed.

Mr. Harry Smith, in the Analyst for June, 1896, describes a method based on the oxidation of formaldehyde by an alkaline solution of potassium permanganate. But as he admits that it is not reliable for smaller quantities than 0·01 gramme formaldehyde, and that there are considerable difficulties in its manipulation, I have, at Dr. Rideal's suggestion, endeavoured to apply the well-known reaction of formaldehyde with an ammoniacal solution of silver nitrate quantitatively, the precipitated silver being either weighed, or the residual silver determined volumetrically. In the actual experiments 10 c.c. of an approximately 0·1 per cent. solution of formaldehyde were added to 25 c.c. $\frac{N}{10}$ silver nitrate, 10 c.c. of dilute ammonia (1 of the 0·88 solution to 50 of water) added, and the whole boiled in a conical flask attached to a reflux condenser. The precipitate, after filtration and washing, was ignited and weighed as metallic silver, and as a check the excess of silver was estimated in the filtrate as silver chloride. As the first experiments showed that the reduction was incomplete after boiling for half an hour, the boiling was continued for four hours. In order to ascertain if any loss took place during boiling, a duplicate determination was made, in which a bottle with tied-down stopper, heated in a water-bath, was employed. The actual results obtained were in the first case 0·01038 gramme formaldehyde, and in the second 0·0104 gramme formaldehyde, consequently there was practically no loss.

In the calculation, as one molecule of CH_2O reduces two molecules of Ag_2O, the weight of the precipitated silver multiplied by the factor 0·0694, gives the weight of the formaldehyde, and 1 c.c. of $\frac{N}{10}$ silver nitrate corresponds to 0·0007495 gramme formaldehyde; it is therefore possible to estimate extremely small quantities by this process.

The following are some duplicate results obtained:

	Percentage CH_2O.	
Approximately 0·1 per cent. solution	(i) 0·1038	(ii) 0·104
Samples of milk-formalin, No. 1	(i) 1·09	(ii) 1·07
,, ,, ,, ,, 2	(i) 0·98	(ii) 0·96
,, ,, ,, ,, 3	(i) 1·02	(ii) 1·02

Incidentally the above results show that the dilute "formalin" supplied for preserving milk which was employed in the experiment is fairly constant in strength.

ON THE SEPARATION AND ESTIMATION OF FORMIC ALDEHYDE.

By Norman Leonard, B.Sc., and Harry M. Smith.

(*Read at the Meeting, December* 2, 1896.)

Some months ago one of us (Analyst, xxi., 148) described a process for the estimation of formic aldehyde, depending upon the oxidation of that substance into formic and carbonic acids by potassium permanganate in alkaline solution. This process we have endeavoured, according to an intention stated in the paper referred to, to apply to the estimation of formic aldehyde in milk, and in doing so we have obtained certain results which seem worthy of notice.

It was of course necessary, in the first place, to ascertain whether pure milk yielded, when distilled, any substance capable of reducing alkaline permanganate Examination of four different samples showed that the distillate from fresh milk exerts practically no such reducing action. The distillate from 100 c.c. of milk required, in fact, scarcely more permanganate than is necessary to give the colour indicating the end-point of the titration. Milks which had been kept several days, still more sour and decomposed milks, yielded appreciable amounts of organic matter on distillation. The process is therefore only applicable to fresh milk.

Several experiments were made on the distillation in a current of steam of milk and aqueous solutions, to which a known amount of formic aldehyde had been added. The recovery of the aldehyde was, however, slow and unsatisfactory. We found, for example, on thus distilling 100 c.c. of liquid containing 0·01 per cent. of formic aldehyde, that the first 50 c.c. of the distillate contained only one-fourth of the total amount of aldehyde added, while less than three-fourths had passed over even when 300 c.c. of distillate had been collected. Simple distillation, though rather more troublesome, was therefore resorted to. The results of three experiments with milk are here tabulated. In each case 100 c.c. of milk was distilled, the distillate being collected in fractions of 20 c.c., each of which was titrated with permanganate solution. The residue remaining in the retort was examined for formic aldehyde by Hehner's test. In the second and third experiments, the liquid was rendered acid and alkaline by the addition of a few drops of dilute sulphuric acid and of caustic soda solution respectively:

	Neutral. Grammes CH_2O.	Acid. Grammes CH_2O.	Alkaline. Grammes CH_2O.
First 20 c.c.	·0015	·0026	·0011
Second 20 c.c. ...	·0006	·0019	·0004
Third 20 c.c.	·0002	·0011	·0003
Residue...	none	present	none
Aldehyde added	·0090	·0090	·0094

It will be noticed that the presence of acid greatly facilitates the expulsion of the aldehyde, while that of alkali tends to retard it. Moreover, the aldehyde was only detected in the residue of the second experiment, in which acid was added. These facts indicate, perhaps, the formation of compounds of the aldehyde with some constituent of the milk, such compounds being broken up, or their formation prevented, by addition of acid.

In these experiments we were somewhat struck by the slowness with which the formic aldehyde, commonly regarded as a very volatile body, distilled over, and we therefore carried out three similar distillations of *aqueous* solutions of the aldehyde. The results are given below. The aldehyde contained in the residue remaining in the retort was in each case determined :

	Neutral. Grammes CH_2O.	Acid. Grammes CH_2O.	Alkaline. Grammes CH_2O.
First 20 c.c.	·0024	·0023	·0026
Second 20 c.c.	·0021	·0018	·0020
Third 20 c.c.	·0017	·0017	—
Fourth 20 c.c.	·0011	·0012	—
Residue...	·0005	·0006	·0031
Total	·0078	·0076	·0077
Aldehyde added	·0078	·0078	·0078

The presence of acid or alkali is here seen to exert little or no influence upon the rate of distillation ; and it appears that the aldehyde is not completely expelled from its solutions by anything short of distillation to dryness. This result might perhaps have been anticipated if we may reason by analogy from the results obtained by Duclaux, and also by Richmond, in the distillation of the fatty acids of the acetic series.

Our experiments lead us to conclude that :

(1) The distillate from fresh milk exerts no appreciable action on alkaline permanganate, and can therefore be used for the estimation of formic aldehyde, the absence of other volatile organic bodies being assumed.

(2) The separation of formic aldehyde from milk by distillation is facilitated by acidulation with sulphuric acid. Under these conditions, the first 20 c.c. of distillate from 100 c.c. of milk will contain about one-third, and the first 40 c.c. about one-half, of the total amount of aldehyde present.

(3) The fact, which has often been adverted to, that the formalin contained in the distillate from milk does not represent the whole of that originally added, is to a great extent explained by the behaviour of solutions of formic aldehyde on distillation, and is only partially due to any specific action of the aldehyde on the constituents of milk.

Discussion.

The President thought it would be interesting if the authors of the first paper could furnish any observations upon the application of their process to milk and other products. So far the paper only dealt with the estimation of formaldehyde in aqueous solution.

In the second paper it seemed to be implied that there was a combination between the formaldehyde and the milk. It was a known fact that formaldehyde polymerized on heating, and it might be that, during the operation of distilling, the formaldehyde polymerized into a molecule more condensed, and more difficult to decompose, than the original molecule.

Mr. Hehner, alluding to a paper which he had some time previously read before

the Society, describing the method which had been referred to as his, said that many people had found difficulty, at first, in obtaining the special reaction with sulphuric acid in the case of milk containing formaldehyde. A suggestion made by one of the authors of the second paper had led to this difficulty being overcome. The suggestion related to the addition of a trace of ferric chloride, which greatly facilitated the reaction.

Apart from the difficulty of distilling formaldehyde from aqueous solution, there was no doubt that formaldehyde combined with certain albuminous substances. He had no experience of its action upon casein, but it certainly combined with albumin to yield a substance which did not coagulate by heat. A solution of albumin containing formaldehyde could be evaporated to dryness, the residue left being completely soluble in water. A process involving this principle had lately been patented. The inventor's conclusion was that two molecules of the albumin were condensed by formaldehyde, the resulting compound being a methylene derivative albumin.

If this conclusion were correct, it would explain why it was not possible to recover the whole of the formaldehyde added to an albuminous substance like milk, since a portion of it would have undergone change in forming the methylene derivative.

Mr. RICHMOND said that almost all organic compounds containing the $-NH_2$ (or $=NH$) group condensed with formaldehyde in the same manner as albumin. Aniline, for instance, gave a compound which was known by the name of anhydro-formaldehyde-aniline, $C_6H_5NH_2 + CH_2O = CH_2NC_6H_5 + OH_2$.

The figures given by Messrs. Leonard and Smith, in relation to fractional distillation, showed that formaldehyde conformed to the law which he had worked out for the volatile acids of the acetic series.

It was curious that higher results should be obtained from acid solutions. The substance liberated by the acid was probably not formaldehyde, but some substance which distilled at about the same rate as water. It would be interesting if some knowledge could be obtained as to the exact nature of this substance, as it would probably throw some light on the changes taking place when formaldehyde and milk were mixed and kept for some time.

Mr. CASSAL said there was no doubt that the addition of formaldehyde to milk profoundly affected the casein. He had made experiments with a view to finding whether the casein was subsequently soluble, and would submit to peptic digestion, and had found that the treatment of milk with the formaldehyde solution in the quantity prescribed by the vendors made the milk indigestible. It curdled into a condition of practical insolubility to peptic digestion. A similar effect was produced on gelatin, which was converted, by weak solutions of formaldehyde, into a hard material resembling brittle horn.

With regard to the separation of formaldehyde from milk, there could be no doubt that, as Mr. Hehner had said, it would be impossible to recover the whole amount added to a sample of milk. It was perfectly obvious that if the formaldehyde entered into combination, something must be done to liberate it.

He would like to ask if Messrs. Leonard and Smith had tried repeated distillations by steam. He understood there was only one continuous distillation in their process.

Some time previously Mr. Bevan had called attention to the fact that an increase in the weight of the residue obtained in milk analyses was found when formaldehyde had been added. As a matter of fact, if a solution of formaldehyde were allowed to evaporate spontaneously, a solid substance remained. How far that might account for what Mr. Bevan found he did not know. He believed, however, that complete reproduction of the formaldehyde could be arrived at by means of repeated distillation with water.

Mr. RICHMOND asked to be allowed to submit to Mr. Cassal a question regarding the indigestibility of casein, viz., whether Mr. Cassal had attempted to prove that the undigested casein was not the well-known dyspeptone. It was a matter of common knowledge that, on submitting casein to peptic digestion, especially in milk which contained a large quantity of lime, an insoluble substance in combination with lime was formed. Had Mr. Cassal made any comparative experiments before and after the treatment with formaldehyde? He, in conjunction with Mr. Boseley, had raised the same question with regard to the experiments of Weigle and Merkel on the same lines as Mr. Cassal.

Mr. CASSAL said that his experiments were carried out side by side, the one with milk containing formaldehyde, the other without. He had not done more than this.

Mr. HEHNER said it was a curious fact that the compound formed by the action of formaldehyde on albumin was eminently digestible. He had seen the results of experiments, which he had every reason to believe were carefully performed, showing that the compound was more digestible than ordinary albumin.

Mr. CASSAL pointed out that Mr. Hehner had said that only a certain amount of the formaldehyde entered into combination, and this combination he considered was somewhat theoretical; consequently there would still be a definite proportion of formaldehyde remaining. He suggested that it was to this extra amount that the preservative effect was due, and also the injurious effect which he referred to

Mr. BEVAN said that, considering the large quantity of casein present in milk, and the small quantity of formaldehyde usually added, it was surprising that any of the latter was obtained at all.

Mr. LEONARD, referring to the President's remarks, said that he understood the product of polymerization was readily converted back into the aldehyde by warming with hot water.

In reply to Mr. Cassal, in the case of the steam distillation, a current of steam was passed into the milk, the operation being continued for several hours. It was not repeated, but was a single continuous operation.

The PRESIDENT, in returning the Society's thanks to the authors of the two papers, remarked upon the important part which formaldehyde played in organic nature, instancing its effects upon gelatin, which had been applied in the practical processes of photography, and its quite different action, which Mr. Hehner had pointed out, in relation to albumin. There were many points requiring elucidation in connection with the subject, which offered an encouraging field of research for young investigators.

NOTES ON PRUSSIAN BLUE.

By Frank H. Leeds, F.I.C.

(*Read at the Meeting, December* 2, 1896.)

A study of the figures given by Messrs. Parry and Coste (Analyst, xxi., 225) as representing the composition of commercial Prussian blue, and more especially the manner in which the authors use the term "pure" blue in its chemical as opposed to its trade significance, have made it seem worth placing on record the experience gained of this substance in the laboratory of a printing-ink works, where the pigments employed have to undergo a more rigorous examination than is perhaps necessary in the case of oil paints.

In the manufacture of printers' ink the material known to chemists as "Prussian" blue is used for two distinct purposes: (1) in the production of a blue ink to be sold as such, and (2) in the preparation of the better qualities of black ink as a means of correcting the invariable brown shade of lamp-black. In this trade Prussian blue is met with under four different names: bronze, pure, Chinese, and "Prussian" blue; but although all of these are equally pure from the laboratory point of view, each variety possesses, when ground in "varnish," a distinct shade of colour, consequently they rank, from the printers' standpoint, as four individual pigments. Bronze blue is the basis of that dark-blue ink so often seen on small advertisements and the covers of magazines, which is specially valued for the coppery lustre, or bronze, it possesses when viewed at the proper angle, more particularly when the impression has been made by lithography. Pure blue yields almost the same shade of ink, but devoid of bronze, and it is therefore seldom used except in making black inks. Both kinds have much the same appearance when in the lump or powder, and it is exceedingly difficult to judge the amount of bronze any ink will have from a mere inspection of the dry pigment. In fact, the material having the most pronounced coppery lustre is usually known as Chinese blue; but this, when ground in oil, produces a brighter and slightly paler cast of blue, almost devoid of bronze. "Prussian" blue is not so often met with; it yields a much darker, duller shade of ink, more resembling indigo as a water-colour pigment, and some samples contain a considerable excess of basic ("extra-radicle") iron. Both pure and bronze blues may be obtained in several slightly different shades, varying from the green to the violet side of blue itself.

A regular examination, extending over several years, of the bronze and pure blues supplied by the leading manufacturers has shown that this pigment varies in composition even less than the samples quoted by Messrs. Parry and Coste; but at the same time it has amply proved that the substance may be accurately valued—apart from the question of shade proper—according to the proportion of acid ("intra-radicle") iron it contains. With the exception that no nitrogen estimations were made, the samples were analysed in the manner given in the paper referred to, but as alumina was almost invariably found to be present in the basic iron precipitate, and titrations showed that it was practically constant in amount, it was usually returned simply as Fe_2O_3, both acid and basic iron solutions being always oxidized with nitric acid before the final precipitation with ammonia. Occasionally a sample containing

up to 20 per cent. of an inert white base is offered under the name of "pure blue"; but this must be regarded either as a mis-description or an adulteration, for such a material is well known under its correct designation of "Antwerp" blue. The average figures obtained on the analysis of the best qualities of these pigments, after drying at 100° C., are given below, and from these it would appear that the previous authors' generalizations are perfectly correct, but that their requirement of 30 per cent. of total iron should be increased to 34 per cent., or preferably that at least 16 per cent. of acid iron should be insisted on.

Pigment.		Basic iron per cent.	Acid iron per cent.
Genuine blues: (bronze, pure, or Chinese).	maximum	19·14	17·16
	minimum	18·37	15·35
	mean	18·90	16·48
"Prussian" blue	,,	22·83	10·01
Adulterated "pure" or Antwerp blue	,,	13·41	12·84

DISCUSSION.

Mr. J. H. COSTE expressed satisfaction at the extent to which, so far as could be gathered from the portion of the paper read, the results obtained by Mr. Parry and himself had been corroborated by Mr. Leeds, whose experience on the subject was much greater than their own.

In their paper they had made some remarks on the appearance of the samples they had analysed, some of which had, in the lump, a bright, coppery appearance, while others, though having a glossy conchoidal fracture, were rather bituminous in appearance. When ground up, however, the samples possessing the bright lustre were by no means the darkest blues, and he thought that was what Mr. Leeds stated. The bright coppery blues, as No. III. in our tables, possessed when ground a much finer colour than the darker, more bituminous colours, as, indeed, did the granular, non-lustrous blues. It was a noteworthy fact that the darker samples contained *alkaline* ferrocyanide.

They considered that the nitrogen determination was a very simple, and at the same time sufficiently accurate, method of estimating, for ordinary painters' purposes, the value of samples of Prussian blue. It could be made very quickly, possessing in this respect a distinct advantage over the determination of the total iron. In the case of pigments ground in oil the percentage of nitrogen had been found to afford a ready means of arriving at a very good idea of the actual quantity of Prussian blue that might be present.

ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

FOOD AND DRUGS ANALYSIS.

The Detection of Alum in Bread. J. Vanderplanken. (*Ann. d. Ph. Louvain*; through *Zeit. Nahr. Unters.*, Vienna, 1896, x., 316.)—In order to avoid the effect of the acidity of old meal or of sour bread on the logwood test, the author recommends the following method: From 10 to 20 grammes of the bread are triturated into a

paste with water, some sodium chloride (free from alkali) added, and then, after the addition of 10 drops of freshly prepared logwood tincture, 5 grammes of pure potassium carbonate gradually mixed in. After being well mixed, the whole is washed with 100 c.c. of water into a beaker, and allowed to settle. After a few minutes the supernatant liquid shows a reddish violet tint when alum is absent, and a grayish blue to a deep blue when it is present. The reaction is capable of detecting 1 gramme of alum in a kilogramme of bread. C. A. M.

The Estimation of Starch in Sausages. J. Mayrhofer. (*Forsch. Ber.*; through *Zeit. Nahr. Unters.*, Vienna, 1896, x., 331, 332.)—The method hitherto employed for the estimation of meal and other starch-containing substances in sausages has been the inversion of the starch present and determination of the sugar produced. Since this, in the author's opinion, is too tedious, and often unsatisfactory, he brings forward in its place the following simple process: The substance under examination is heated on the water-bath with alcoholic potash (about 8 per cent.), which in the case of pure sausages dissolves almost everything except a little cellulose. The solution is diluted with warm alcohol, to prevent gelatinization, and filtered through a paper or asbestos filter. The insoluble residue, containing the starch when present, is washed with alcohol until the washings are no longer alkaline, then treated with *aqueous* potash, and the starch solution made up to a definite volume. On adding alcohol to an aliquot portion, the starch falls as a flocculent precipitate, which rapidly subsides. This is collected on a weighed filter, washed with alcohol and ether, dried and weighed.

In order to avoid a determination of the ash, it is advisable to make the starch precipitation from a weak acetic acid solution instead of from an alkaline solution, since the acetate formed from the potassium carbonate in the starch is readily soluble in alcohol. By this means the starch is obtained quite free from ash.

From 60 to 80 grammes of the sausage should be taken for the determination, since the starch is usually not regularly distributed throughout the mass.

As a test of the accuracy of the method, mixtures were made of about 60 grammes of sausage, with varying amounts of pure potato starch dried at 100° C., and analysed as described above, with the subjoined results:

Starch added. Gramme.	Starch found. Gramme.
0·058	0·056
0·133	0·132
0·097	0·097
0·098	0·098
0·341	0·340
0·260	0·255

C. A. M.

A New Method of estimating Casein in Milk. G. Denigès. (*Bull. Soc. Chim.*, 1896, 1116-1126.)—This is an application of the author's general process for the estimation of mercury (ANALYST, xxi., 303). On adding to a solution of

potassium cyanide (equivalent to $\frac{N}{10}$ silver nitrate) 10 cc. of an $\frac{N}{10}$ solution of mercuric potassium iodide, 10 cc. of ammonia, and 100 cc. of water, and running in $\frac{N}{10}$ silver nitrate solution, the amount of the latter required to produce a permanent turbidity will not be 10 c.c., but only 4·8 c.c. If, then, the casein in a milk be precipitated by the addition of $\frac{N}{10}$ mercuric potassium iodide and acetic acid, and the excess of mercury determined with silver nitrate, the amount of mercury combined with the casein will be equivalent to $(q - 4{\cdot}8)$ c.c. of $\frac{N}{10}$ silver nitrate.

Twenty-five c.c. of the milk are placed in a 200 c.c. flask, 20 c.c. of $\frac{N}{10}$ mercuric potassium iodide and 2 c.c. of glacial acetic acid added, and the liquid made up to the mark, and filtered. To 100 c.c. of the clear filtrate, 10 c.c. of potassium cyanide solution (equivalent to $\frac{N}{10}$ silver nitrate) are added, and the liquid titrated with $\frac{N}{10}$ silver nitrate, the results being expressed in tenths of a c.c. after the deduction of the constant 48 $(q - 48)$.

The $\frac{N}{10}$ mercuric potassium iodide solution is prepared by dissolving 13·55 grammes of mercuric chloride and 36 grammes of potassium iodide in distilled water, and making up to a litre.

In order to obtain the value of $(q - 48)$, a large number of determinations were made with different kinds of milk, a simultaneous estimation of the casein being made in every instance by the Adam-Roux process, and it was found that $(q - 48)$ did not vary in exact proportion with the amount of casein in the milk, but that for milks rich in casein it was less than the number which gave the real amount of casein per litre, and was greater for poor milks. This variation is plotted in a curve, in which the quantities of casein per litre of milk, x, form the ordinates, and the corresponding values of $(q - 48)$ the abscissæ. From the curve the author has established the following table of relation between x and the values of $(q - 48)$:

$\frac{N}{10}AgNO_3$ in $\frac{1}{10}$ c.c.'s.	Casein. Grammes per litre.	$\frac{N}{10}AgNO_3$ in $\frac{1}{10}$ c.c.'s.	Casein. Grammes per litre.	$\frac{N}{10}AgNO_3$ in $\frac{1}{10}$ c.c.'s.	Casein. Grammes per litre.
0	0	16	14	32	32
1	1	17	15	33	33·50
2	1·7	18	16	34	35
3	2·5	19	17	35	37
4	3	20	18	36	39
5	3·75	21	19	37	40·50
6	4·50	22	20	38	42·75
7	5·50	23	21	39	45
8	6·50	24	22·25	40	47
9	7·15	25	23·50	41	49
10	8	26	24·75	42	51·50
11	9	27	26	43	54
12	10	28	27	44	57·20
13	11	29	28	45	60
14	12	30	29·25	46	62·50
15	13	31	30·75		

In the following tabulated determinations, the results, calculated from this

volumetric method, are compared with those obtained by the gravimetric process of Adam-Roux:

Milk.	$\eta - 48$.	Casein. Weighed. Grammes.	Casein. Calculated. Grammes.	Difference. Grammes.
Cow's, whole	33·5	34·25	34	+0·25
diluted to $\frac{1}{2}$	19	17	17	0
,, ,, $\frac{1}{4}$	10·50	8·50	8·50	0
Goat's, whole...	35	37	37	0
diluted to $\frac{1}{2}$	20·50	18·50	18·50	0
,, ,, $\frac{1}{4}$	11·50	9·50	9·25	+0·25
,, ,, $\frac{1}{8}$	6·50	5	4·62	+0·38
Sheep's, whole	46·50	65	65	0
diluted to $\frac{4}{5}$	42	51·50	52	+0·50
,, ,, $\frac{3}{5}$	36	39	39	0
,, ,, $\frac{2}{5}$	27	26	26	0
,, ,, $\frac{1}{5}$	15	13	13	0
Ass's, whole	21	19	19·10	−0·10
diluted to $\frac{1}{2}$	11·50	9·50	9·55	−0·05
,, ,, $\frac{1}{4}$	6·50	5	4·77	+0·23
Mare's, whole...	25	23·50	23·60	−0·10
diluted to $\frac{1}{2}$	14	12	11·80	+0·20
,, ,, $\frac{1}{4}$	7·50	6	5·90	+0·10
Woman's, whole	13	11	11	0
diluted to $\frac{1}{2}$	7	5·50	5·50	0
Cow's (boiled), whole ...	36·50	39·75	40	−0·25
diluted to $\frac{1}{2}$	21·50	19·50	20	−0·50
,, ,, $\frac{1}{4}$	12	10	10	0
,, ,, $\frac{1}{8}$	6 50	5	5	0
Cow's (sterilized), whole ...	35·50	37·50	37·90	−0·40
diluted to $\frac{1}{2}$	20·50	18·75	18·95	−0·20
,, ,, $\frac{1}{4}$	11·50	9·50	9·47	+0·03
Cow's (condensed), in presence of cane-sugar, whole ...	25·50	24·12	24·50	−0·38
diluted to $\frac{1}{2}$	14	12	12·25	−0·25
,, ,, $\frac{1}{4}$	7·50	6	6·12	−0·12
Goat's (boiled), whole ...	46	62·50	62·50	0
diluted to $\frac{1}{2}$	31	30·75	31·25	−0·50
,, ,, $\frac{1}{4}$	17·50	15·50	15·62	−0·12
,, ,, $\frac{1}{8}$	10	8	7·81	+0·19
,, ,, $\frac{1}{16}$	5	3·75	3·90	−0·15

From these and other experiments, the author concludes that the method is applicable to all possible cases with the exception of milks containing formaldehyde, which exercises a disturbing influence.

Without having recourse to the table or the curve, the corrections may be made simply in the following manner: When $(q - 48) = a$, lies between 9 and 24, $x = a - 2$, that is to say, the constant 2 must be subtracted from the number of tenths of a c.c. of $\frac{N}{10}$ silver nitrate used. When a lies between 25 and 32 inclusive,

$$x = a - \frac{32 - a}{4} = \frac{5a - 32}{4}.$$

And where a is above 33, $x = 2a - 33$. In cases where a is above 37, it is advisable to dilute the milk by one-half, since the method is more sensible with dilute than with concentrated solutions of casein.

It is claimed that this method can be carried out in five minutes, whilst the gravimetric method requires three or four hours. The fact that the caseins from the different milks, although dissimilar in appearance, behave in an absolutely identical manner towards mercuric potassium iodide points to a great similarity, if not complete identity, between them all. The author is examining the application of this process to the determination of other albuminoid bodies. C. A. M.

The Chemical Examination of Cheese. A. Stutzer. (*Zeit. anal. Chem.*, 1896, xxxv., 493-502.)

Ash and Mineral Matter.—From 10 to 15 grammes of the cheese are burnt (preferably in a muffle-oven) in a platinum basin. The weighed ash is dissolved in 250 c.c. of water, and aliquot portions used for the determination of chlorine, calcium and phosphoric acid.

Water.—A weighed quantity of the cheese is mixed with washed, ignited, and sifted quartz sand. For most cheeses the proportions of 100 to 400 grammes of sand are satisfactory, but with very rich cheeses 500 grammes of sand are taken. This sand mixture is used in all the estimations. For the determination of the water, an amount of the mixture corresponding to about 3 grammes of cheese is dried to constant weight in the water-oven.

Fat.—The dry residue from the water-determination is extracted for twenty-four hours with water-free ether, which has been treated with sodium. The author considers that there is less chance of error by this method than in that of Fleischmann, which is frequently employed.

Nitrogen.—I. *Total Nitrogen.*—Ten grammes of the sand-mixture are analysed by Kjeldahl's method. The author considers it preferable to remove the fat first, but mentions that Bondzynski (*Landw. Jahrbuch der Schweiz*, 1894, 197) obtained bad results when doing this.

II. *Applicability of Copper Hydrate to the Precipitation of Albuminoids.*—While copper hydrate can often be used to separate albuminoids from amido compounds

and ammonium salts, the author showed in a former communication (*Zeit. anal. Chem.*, xxxi., 505) that it only partially precipitated pancreas peptone (Kühne's peptone). Thinking that cheese peptone might differ in this respect from flesh peptone, he extended his experiments to the former; but having experienced the same difficulty, he concluded that copper hydrate was not a suitable precipitant for cheese albuminoids, since the latter contained pancreas peptone (Kühne's peptone).

III. *Phospho-tungstic Acid as a Precipitant.*—Bondzynski's conclusions (*loc. cit.*) as to the value of phospho-tungstic acid as a separating agent are confirmed. By its means the caseins and albumins and their primary decomposition products (including the albumoses and peptones) are separated from the worthless decomposition products—phenyl-amido-propionic acid, tyrosine, leucine, and other amides—as well as from the ammoniacal compounds.

The substances belonging to the first group may be further divided into (*a*) Indigestible nitrogenous matters, (*b*) Albumoses and peptones soluble in boiling water, and (*c*) Caseins and albuminates insoluble in boiling water.

IV. *Nitrogen in the form of Ammoniacal Salts.*—An amount of the sand-mixture corresponding to 5 grammes of the cheese is mixed with 200 c.c. of water, and the ammonia distilled after the addition of barium carbonate. Magnesia and magnesium carbonate (which usually contains some magnesia) cause a partial decomposition of the amides.

V. *Nitrogen in the form of Amides.*—This is taken to be the nitrogen belonging to those compounds in the cheese which are not precipitated by phospho-tungstic acid, and which are not ammoniacal compounds. An amount of sand-mixture corresponding to 5 grammes of cheese is mixed with 150 c.c. of water, and well shaken for fifteen minutes in a closed vessel. After standing for fifteen hours at the ordinary temperature, 100 c.c. of dilute sulphuric acid (1 vol. : 3 vol. water) are added, and phospho-tungstic acid so long as a precipitate results. The liquid is filtered, the precipitate washed with dilute sulphuric acid until the filtrate amounts to 500 c.c., and the nitrogen is determined in 200 c.c. of this. By deducting from the amount that previously found as ammoniacal nitrogen, the nitrogen present in the form of amides is obtained.

VI. *Indigestible Nitrogenous Substances.*—The fresh mucous membrane of pigs' stomachs (preferably six) is cut into small fragments and mixed with water and hydrochloric acid in a wide-necked flask in the proportion of 5 litres of water and 100 c.c. of 10 per cent. (by weight) hydrochloric acid to each stomach. At the same time, 2½ grammes of thymol dissolved in alcohol are added as a preservative. The mixture is left for twenty-four hours, with occasional shaking, and then filtered through flannel, coarse paper and fine paper successively. If necessary, the amount of hydrochloric acid in the extract is brought to exactly 0·2 per cent. As thus prepared, the gastric juice remains unaltered for months.

Sand-mixture containing 5 grammes of cheese is deprived of its fat by extraction with ether, mixed with 500 c.c. of the gastric juice in a beaker, and the mixture warmed for forty-eight hours in a thermostat at 37°–40° C. At intervals of about two hours, 5 c.c. of 10 per cent. hydrochloric acid are added, until the acidity of the whole reaches 1 per cent. The liquid is then filtered through paper or asbestos, the

residue washed with water, and the nitrogen contained in it determined. According to Kühne (*Landwirths. Versuchsstat.*, xliv., 194), subsequent treatment with alkaline pancreatic extract is unnecessary when the acid pepsin digestion has been continued for forty-eight hours.

VII. *Nitrogen in the form of Albumose and Peptone.*—A weighed quantity of sand-mixture, containing 5 grammes of cheese, is extracted by boiling with successive portions (100 c.c.) of water, the liquid made up to 500 c.c. and filtered, and 200 c.c. of the clear filtrate, mixed with an equal volume of dilute sulphuric acid, and precipitated with phospho-tungstic acid.

Qualitative Test for Pancreas Peptone.—A portion of the hot-water extract is concentrated by evaporation, saturated with zinc sulphate (ANALYST, xxi., 16), and filtered. Concentrated sodium hydrate is added to the filtrate until the zinc hydrate dissolves, when a few drops of a 1 per cent. solution of copper hydrate are added; the biuret reaction points to the presence of pancreas peptone. The quantitative estimation is carried out as described by the author in his former communication (ANALYST, xx., 249), using Bömer's zinc sulphate modification (ANALYST, xxi., 16).

VIII. *Caseins and Albumins.*—The nitrogen present in these substances, which are insoluble in boiling water, is obtained by subtracting from the total nitrogen the amounts found in V., VI., and VII. It is not advisable to use the residue from the extraction in VII. for the purpose, since the large amount of sand causes violent bumping in the Kjeldahl flask.

IX. *Separation of the Caseins and Albumins Digestible with difficulty from those readily Digestible.*—Cheese contains only small quantities of completely indigestible nitrogenous substances, and it is therefore useful to determine the comparative digestibility of the caseins and albumins. For this purpose a process of "interrupted digestion" is employed. In order to obtain comparable results, care is taken to have constant (1) the amount of nitrogen in the form of insoluble, but digestible, casein and albumin; (2) the amount of gastric juice; and (3) the acidity of the liquid, and the temperature and duration of the digestion.

In each experiment, so much of the sand-mixture is taken as contains 0·15 gramme of nitrogen in the form of insoluble, but digestible, casein and albumin, to which is added 150 c.c. of the gastric juice, with 343 c.c. of water and 7 c.c. of 10 per cent. hydrochloric acid. The acidity of the total liquid ($\frac{1}{2}$ litre) is exactly 0·20 per cent., the temperature is maintained at 37° to 40° C., and the duration of the digestion is thirty or sixty minutes. The liquids are warmed to 40° C. before being measured and after mixing. At intervals of five minutes during the digestion the liquid is stirred with a glass rod; and at the conclusion the total liquid is placed in two large folded rapid filters, and a portion of the filtrate passing through in the first five minutes taken for the determination of the nitrogen. From the result, a deduction must be made for the nitrogen contained in the gastric juice, and for the nitrogen in the cheese dissolving without the aid of the gastric juice (amide, ammoniacal, albumose, peptone).

The following results obtained in the examination of three varieties of cheese illustrate this investigation:

	Camembert. Per cent.	Swiss. Per cent.	Gervais. Per cent.
Water	50·90	33·01	44·84
Fat	27·30	30·28	36·73
Fat-free organic matter	18·66	31·41	15·48
Ash	3·14	5·30	2·95
The ash contained—			
Calcium	0·03	1·56	0·14
Phosphoric acid	0·76	0·82	0·23
Sodium chloride	2·21	1·56	0·76
Total nitrogen	2·900	5·072	1·923
Nitrogen as ammonia	0·386	0·188	0·031
,, amides	1·117	0·459	0·099
,, albumoses and peptones	0·885	0·435	0·298
,, indigestible matter	0·115	0·119	0·166
,, casein and albumin	0·397	3·871	1·329
Percentage of casein and albumin dissolved by gastric juice—			
In 30 minutes	100	68	52
,, 60 ,, ...	100	91	75
100 parts of nitrogen were present in the following forms—			
As ammonia	13·0	3·7	1·6
,, amides	38·5	9·0	5·2
,, albumose and peptones	30·5	8·6	15·5
,, indigestible matter	4·0	2·4	8·6
,, casein and albumin	14·0	76·3	69·1

C. A. M.

Analysis of a Spurious Roasted Coffee. M. Maljean. (*Jour. Pharm. Chim.*, 1896, 352-355.)—This product, which was sold by a Marseilles firm under the name of "roasted grains," closely resembled the genuine coffee-bean in appearance and form, and, although close inspection could detect the difference, might easily escape notice when mixed with true coffee. The grains were of a maroon colour, very hard, and very much more regular than coffee-beans. On close examination a seam could be observed in all of them exactly in the middle, and, as a rule, not reaching to the edges, which evidently showed that the grains had been formed by pressure in a mould.

A mixture of the false grains with true coffee could be easily detected by placing it in water, when, owing to their greater density, the former sank.

On being boiled with water for thirty minutes the false coffee did not disintegrate, but only softened; but on being left for several hours in contact with a solution of sodium hydrate, it swelled up, and finally formed a paste. This did not occur with true coffee.

The analysis of the grains (not dried) gave the following results:

	Per Cent.
Water	10·00
Fat	1·90
Nitrogenous matter	13·12
Aqueous extract (20·25), containing Sugar	1·87
Aqueous extract (20·25), containing Gum	5·20
Aqueous extract (20·25), containing Tannin	0·18
Aqueous extract (20·25), containing Other soluble matter ...	13·00
Starch...	47·63
Caffeine	0·00
Cellulose	3·70
Ash	3·40

The ash was neutral, and contained:

	Per Cent.
Part soluble in water	10·30
,, insoluble ,, ...	89·70
Phosphoric acid	23·814
Sodium chloride	0·20
Silica	14·70

The fat was yellow and odorless, and had a consistence similar to that of the fat from flour.

The author came to the conclusion that the false grains consisted of an intimate mixture of meal with bran, and an unknown oleaginous seed of little value, and that the mixture was roasted and subsequently agglutinated with gum or dextrin in special moulds. The gluten in the flour employed would also help to give to the grains their great cohesion. C. A. M.

A Contribution to the Study of Maté. P. Macquaire. (*Jour. Pharm. Chim.*, 1896, 346-350.)—It is shown that maté is a plant which only yields its soluble matter with great difficulty to hot water, and that even after many successive decoctions of twenty minutes each with water at 80° C., a large amount of the soluble substances are left in the residue. The first extract, however, contains nearly the whole of the alkaloid present, whilst the tannin is also found almost entirely in the first decoctions. The subsequent extracts consist chiefly of various resinous bodies, only slightly soluble in water, but readily soluble in chloroform and ether. On treatment of maté with boiling chloroform, the author obtained 10·83 per cent. of a resinous body, only slightly soluble in water and acids, but very soluble in alkalies.

After preliminary trials as to the best method of estimating the caffeine, the following was adopted: An excess of ammonia was added to the aqueous extract of the maté, and the mixture shaken with successive portions of chloroform in a separating funnel. The chloroform extracts were evaporated to dryness, the residues taken up with hot dilute sulphuric acid (1 : 10), the solutions filtered, evaporated to dryness, and the residue again taken up with chloroform, which on being evaporated left the caffeine pure. By this process the author obtained 8·75 grammes of caffeine per kilogramme. He concludes that it is essential to use a large excess of water to dissociate the caffeine from its combination, and in this confirms the statements of

Petit on the estimation of caffeine in tea (ANALYST, xxi., 232). The colour reactions and melting-point of the pure alkaloid (234° C.), and the fact that the melting-points of crystals obtained by fractional crystallization from chloroform were identical, prove that the alkaloid in maté really is caffeine.

The sample of maté examined contained 6·113 grammes of mineral matter per kilogramme, of which 2·82 grammes were soluble in water, and 3·293 grammes insoluble. A considerable quantity of iron was present. C. A. M.

ORGANIC ANALYSIS.

A Study of the Methods for the Determination of Starch. H. C. Sherman. (*School of Mines Quarterly*, 1896, xvii., 356-365.)—The wide distribution of pentosans such as xylan and araban, and their susceptibility to hydrolysing agents, accounts for the high results often obtained by most of the methods for the determination of starch in the presence of other insoluble matter (fibre). In the present paper the author has examined the accuracy of the various methods, with special reference to the pentosans.

The starch solutions were tested in each case by the furfurol test, and when a negative result was obtained it was concluded that no error was introduced from this cause. About 25 c.c. of the solution to be tested were placed in a beaker, with the same volume of 25 per cent. hydrochloric acid, the beaker covered with a watch-glass, and its contents heated to boiling. On introducing a slip of filter-paper moistened with aniline acetate (made by mixing equal volumes of aniline and strong acetic acid) into the escaping vapours, a bright-red coloration was obtained when furfurol was being given off.

The methods examined may be divided into four classes, according to the degree to which inversion is carried before the removal of foreign matter from the influence of the solution. (*a*) Complete hydrolysis to glucose (as in Sachsse's method); (*b*) conversion into maltose and dextrin by ferments (Maercker's and diastase methods); (*c*) conversion to dextrins by acids (Guichard's method); (*d*) solution without chemical change (Baudry's and salicylic acid method). Most of these methods were described in an abstract on this subject in the ANALYST, xx., 17. In Baudry's method the author adopted the modification of heating for one hour on the water-bath instead of boiling.

The salicylic acid method was based on the results of experiments of Baudry's process, but had only the solvent in common with it. From one to three grammes of the finely-ground material were extracted with water, and then heated in a flask with 100 c.c. of water and 0·5 gramme of salicylic acid, in the water-bath till all the starch was rendered soluble (about 15 minutes). The liquid was filtered through muslin, the residue washed with hot water, and an aliquot part of the filtrate taken. To this one-tenth of its volume of 25 per cent. hydrochloric acid was added, and, after being heated for 45 minutes on a sand-bath under a reflux condenser, the glucose estimated in the usual manner. It was proved that in this way all the starch was made capable of being filtered before the pentosans were sensibly attacked.

In the subjoined tables, I. gives the results obtained by the different methods, and II. the results of qualitative tests for pentosans in the final solutions, the figures 1, 2, and 3 indicating the relative intensity of the positive tests. The "pure" starch sample was prepared by repeatedly washing good corn starch with dilute saline solution and water, and drying the final product in the air. It contained: ash, 0·20 per cent.; moisture, 17·36 per cent.; starch, by difference, 82·44 per cent. All the samples were ground to pass through a 60-mesh sieve, and the water-soluble matter extracted in every case before making the analysis.

I.—Percentage of Starch.

Sample.	Malt Method.	Maercker's Method.	Salicylic Acid Method.	Sachsse's Method.	Baudry's Method, modified.	Guichard's Method.
"Pure" starch	82·49	82·39	82·33	82·30	82·33	82·50
Wheat flour	66·55	67·15	66·84	68·35	...	...
Oatmeal	56·23	56·16	56·00	59·01	...	...
Graham flour	55·32	...	55·66	58·63	...	...
Wheat bran	20·97	21·07	21·57	38·82	29·74	31·36
Wheat straw	4·39	4·80	4·41	22·69	5·33	8·93
Corn fodder	0·96	...	0·92	20·13	...	...

II.—Qualitative Tests for Pentoses in Solution.

Sample.	Cold water Method.	Malt Method.	Maercker's Method.	Salicylic Acid Method.	Sacchse's Method.	Baudry's Method.	Guichard's Method.
Wheat flour ...	1	0	0	0	?	...	...
Graham flour ...	?	0	...	0	1	...	...
Wheat bran ...	0	0	?	1	3	2	2
Wheat straw ...	0	0	0	0	3	?	2
Corn fodder ...	0	0	...	0	3	...	...

From these results the author concludes that: (1) The diastase, or malt method, gives satisfactory results, and is not vitiated by any substances known to occur in cereals or grasses. (2) Maercker's and the salicylic acid methods give results fairly comparable with those of the diastase method. (3) No method based on the chemical transformation of starch by acids before removal from other substances present can be accepted as generally applicable. (4) A qualitative test for pentoses in the final solution is a valuable check in working with any of the inversion methods.

C. A. M.

The Estimation of Starch in Cereals. L. Lindet. (*Bull. Soc. Chim.*, 1896, 1163-1164.)—The various methods hitherto employed for the estimation of starch in cereals do not take into account the presence of other carbohydrates, especially sugars. Extraction of sugar with water tends to bring about the action of the diastase which is found even in non-germinated grains, whilst alcohol, though

destroying diastatic action, does not dissolve the gums (Lintner's gum, galactine, levosine). In the proposed method the author dissolves the glutinous envelope surrounding the starch by means of a solution of pepsin, and then extracts the starch by trituration and sifting. About 10 grammes of the ground grains are covered with a solution containing 1·5 c.c. of hydrochloric acid per cent. and 2 per cent. of pepsin, and the flask left for twelve or twenty-four hours, with occasional agitation, at a temperature of from 40° to 50° C. The hydrochloric acid prevents the diastase from acting on the starch without interfering with the digestion of the gluten, and the author's experiments show that under the conditions of the estimation the starch is not hydrolysed by the acid. The contents of the flask are then placed on a hair-sieve (No. 80-100), the sieve tied into a knot, and the knot enclosing the grains triturated in a basin with successive portions of water until no more starch comes through the sieve. The several starch waters are united, some corrosive sublimate or formic aldehyde added to prevent bacterial action, and the starch collected on a weighed filter. The filtration, which is tedious, may be accelerated by adding a small quantity of washed and calcined pumice-stone. The filter is dried at 50° C., and subsequently at 105° C., and weighed. The method is said to give good results with oats, wheat, and barley, as well as with malt, which always contains a certain proportion of sugar. C. A. M.

The Analysis of Commercial Glucose. J. Weiss. (*Zeit. Nahr. Untersuch.* Vienna, 1896, x., 282.)—With reference to Denamur's method of analysis (ANALYST, xxi., 194) the author considers it advisable to obtain more information as to the non-fermentable extract. In a sample described as "maltose syrup, rich in iso-maltose, he found, per 100 grammes,

Water	22·40
Extract	77·60

and in the latter:

Maltose (fermentable sugars)	46·80
Dextrin and substances fermenting with difficulty	19·80
Dextrin	10·00
Inorganic matter (ash)	0·90

C. A. M.

On the Critical Temperature of Fats and Oils. A. von Asbóth. (*Chem. Zeit.*, 1896, xx., 685, 686.)—This paper contains the results of a number of tests on pure butter and margarine which, in the main, confirm Crismer's figures (ANALYST, xx., 209 and 257). The critical temperatures obtained were 12° higher than those of Crismer, owing to the author having used an alcohol of specific gravity 0·8332 (90 per cent. by vol.). Eight samples of perfectly pure butter gave from 111·5° to 115°, while two samples of margarine gave 133° and 133·5° respectively, and a sample of oleomargarine 142°. The greatest difference between butter and oleomargarine was 30·5° (Crismer 30°), and the smallest 27° (Crismer 16°); while the greatest and smallest differences between butter and margarine were 23° and 18° C.

C. A. M.

Detection of Japan Wax in Ox-tallow. C. Dietrich. (*Phar. C. H.*; through *Zeit. Nahr. Unters.*, Vienna, 1896, x., 330, 331.)—Recently Japan wax has been considerably employed to adulterate South American ox-tallow, and to detect the addition the author recommends the following process: On warming 26 grammes of pure ox-tallow with 75 grammes of petroleum spirit, a clear golden solution is obtained, which on cooling remains completely clear, or at most deposits a few crystals. Japan wax, on the other hand, dissolves in petroleum spirit with great difficulty. With an ox-tallow which has been adulterated with Japan wax to the extent of 5 to 10 per cent., the solution, after long warming remains turbid, white, opaque, and emulsion-like; an addition of 1 per cent. caused the solution to remain opalescent.

Another simple test for Japan wax is the borax test. On boiling 0·5 gramme of the fat with 20 c.c. of a saturated solution of borax, and then cooling, the aqueous layer in the case of pure ox-tallow is completely clear and sharply separated from the fatty layer. Should, however, as little as 5 per cent. of Japan wax be present, there is neither a separation nor a clear aqueous layer, but the mass remains as a white opaque emulsion.

These reactions, in conjunction with the determination of the iodine number and critical temperature, will detect the falsification with certainty. C. A. M.

Antipyrine as a Test for Nitrites. M. C. Schuyten. (*Chem. Zeit.*, 1896. xx., 722.)—Schaack has already described a colorimetric process for the estimation of antipyrine, based on the green colour produced by nascent nitrous acid. A solution of 1 part of antipyrine in 100 of 10 per cent. acetic acid may also be employed to detect nitrites, the colour appearing rapidly even in dilutions of 1 : 20,000. The reaction is very characteristic and permanent, for the colour is only affected by oxidising agents such as ferric salts, or by hydrochloric and sulphuric acids, which turn it yellow. By the aid of a series of standard tints the test may be made quantitative; and in the examination of potable waters it should be found to possess considerable advantages over the zinc iodide and permanganate processes, as ordinary organic substances (sugar, alcohol, phenols) have no influence on the colour.

F. H. L.

Detection and Estimation of Nitrites in Water. Barbet and Jandrier (*Jour. Pharm. Chim.*, 1896, 248.)—The hydrochloride of metaphenylene-diamine, commonly used for the estimation of nitrites, has the drawback of being difficult to keep colorless. The authors describe the following method in which it is replaced by resorcin: 0·1 gramme of resorcin is dissolved in 2 c.c. of the water under examination, and 1 c.c. of pure concentrated sulphuric acid added, the acid being allowed to run down the side of the tube so as to avoid an immediate mixture. The colour is first formed at the line of juncture of the two liquids and gradually intensifies. The tube is then shaken gently, care being taken not to raise the temperature too high, and after standing for an hour the colour formed is compared with the tints obtained under precisely similar conditions with solutions containing known quantities of

sodium nitrite. Water containing 0·01 in 100,000 of nitrite of sodium gives a distinct rose coloration after standing for several hours. A colorimetric scale corresponding to the different quantities of nitrites can be established by comparing the results with a solution of cobalt chloride diluted to different strengths with distilled water.

C. A. M.

INORGANIC ANALYSIS.

Estimation of Sulphides in Calcium Carbide. F. J. Pope. (*Jour. Amer. Chem. Soc.*, 1896, xviii., 740-741.)—A weighed quantity of the carbide is placed in an Erlenmeyer flask connected with a wash-bottle containing 150 c.c. of standard lead acetate solution (about decinormal), this in turn being connected with a smaller wash-bottle. Water is carefully introduced through a stop-cock funnel into the flask until there is no further evolution of acetylene, when 25 to 40 c.c. of sulphuric acid (1 : 3) are added, and the whole boiled, the final traces of sulphuretted hydrogen being removed by means of a current of air. The contents of the wash-bottles are filtered, the filtrate made up to 100 c.c., and the amount of unconsumed lead acetate estimated volumetrically (see ANALYST, this volume, p. 269). The difference between the result and the amount of lead acetate started with gives the amount precipitated by the sulphuretted hydrogen, from which the amount of sulphur as sulphide can be calculated.

In a sample of calcium carbide the author found the following percentage of sulphur :

Calcium Carbide taken. Grammes.	Sulphur found. Per cent.
2·4492	3·37
3·1234	3·57

No check was applied to the method, and obviously, besides sulphuretted hydrogen, other substances may be evolved from impure calcium carbide which can remove lead from the solution. The author proposes to investigate the subject further.

C. A. M.

The Separation of Undissolved Phosphates in Mixed Fertilizers. A. P. Bryant. (*Jour. Amer. Chem. Soc.*, 1896, xviii., 491-498.)—Owing to their cheapness mineral phosphates have been largely employed instead of bone as a source of phosphoric acid in fertilizers. To determine the origin of the "insoluble" phosphoric acid the author has made use of the difference in specific gravity between bone and other organic matter and mineral phosphates. The specific gravities of some of the most important constituents found in bone and rock phosphates are :

	Specific Gravity.
Bone and other organic matter, less than	2·0
Gypsum	2·3
Aluminium phosphates, above	2·3
Iron phosphates, about	2·6
Silica	2·65
Calcium phosphates	2·9 to 3·55
Fluorite	3·2

For the separating solution 75 grammes of potassium iodide and 100 grammes of mercuric iodide are dissolved in 350 c.c. of water, and evaporated on the water-bath to a specific gravity of 2·26.

The apparatus required is a separating and an extraction tube. The former consists of two tubes $1\frac{3}{10}$ c.m. internal diameter, one 7 c.m. long, and closed at one end, the other 20 c.m. in length. These are connected by indiarubber tubing, so that the lower portion can be separated by a pinchcock. The extraction tube is $2\frac{1}{2}$ c.m. internal diameter, slightly contracted at one end, and with a rim over which filter-paper and cheese-cloth are tied.

Two grammes of the substance to be examined are transferred to the extraction tube, and washed with from 100 to 225 c.c. of hot water, according to whether water-soluble phosphoric acid is to be estimated or not. The tube and its contents are dried thoroughly in the water oven, and when dry the filter-paper is taken off and all matter carefully removed with a spatula and brush to the separating tube. From 15 to 20 c.c. of the separating solution are added, the tube well shaken and its sides washed down with a jet of the solution. After standing five minutes the lower part of the tube is tapped with the finger to release any light portion carried down with the heavy, whilst the matter on the top is stirred up with a jet of the solution.

The tube is left for an hour, or until the solution is clear, when the rubber connecting tube is clamped, and the lower portion removed, the fingers being protected by rubber tips. The separated portions are filtered, washed, and treated with ammonium citrate solution, and the insoluble phosphoric acid determined in the usual way. That in the "light" portion comes from bone, tankage, or other organic matter, whilst that in the "heavy" is derived from minerals. The author's experiments show that the fertilizer cannot be treated with ammonium citrate solution before separating, and that matter soluble in water must be first removed.

To test the method mixtures were made of prepared fertilizers with Florida and Carolina raw and dissolved rock, the amount of total and insoluble phosphoric acid in each being known, and the following results among others were obtained with a separating solution of specific gravity 2·26, which was found the most satisfactory strength.

PERCENTAGE OF PHOSPHORIC ACID.

No.	Light.		Heavy.			Total.	
	Theoretical.	Found.	Theoretical.	Found.	Left on Paper.	Theoretical.	Found.
7. Mixture ...	1·28	1·38	12·18	11·65	0·12	13·46	13·15
8. ,, ...	1·28	1·63	6·28	6·03	0·06	7·56	7·72
9. ,, ...	1·28	1·18	0·39	0·65	trace	1·67	1·83
10. ,, ...	1·28	1·28	12·18	12·03	0·12	13·46	13·43
11. ,, ...	1·28	1·35	6·28	6·08	0·06	7·56	7·49
15. Prepared mixed fertilizer ...	2·56	2·51	—	—	trace	2·56	2·51
16. Dissolved rock	—	—	0·77	0·70	trace	0·77	0·70

The methods of analysis were those of the Association of Official Agricultural Chemists.

C. A. M.

A Method of Titrating Soluble Iodine Compounds. E. Riegler. (*Zeit anal. Chem.*, 1896, xxxv., 305-307.)—The principle of the method consists in liberating iodine from the iodides by means of iodic acid, taking up the iodine in petroleum spirit, and titrating the excess of iodic acid with decinormal thiosulphate solution. The amount of iodine is calculated from the amount of iodic acid used, $\frac{5}{6}$ of each atom of iodine being due to the decomposed iodide. The equations of the reactions are given in the following abstract. For the preparation of a decinormal solution 17·6 grammes of iodic acid are dissolved in a litre of water.

The details of the estimation are as follow: Several c.c. of the iodide solution, which must not be more concentrated than 1 per cent., are placed in a stoppered separating funnel, an equal volume of decinormal iodic acid solution added, and the funnel well shaken. After the addition of 20 c.c. of petroleum spirit, the shaking is continued until the greater part of the iodine is taken up, the remainder being removed by a fresh portion of petroleum spirit. The aqueous layer is then titrated with decinormal thiosulphate solution, starch solution being used as indicator. The difference between the amount of iodic acid taken and that left, multiplied by the factor $0{\cdot}0127 \times \frac{5}{6} = 0{\cdot}0106$, gives the iodine corresponding to the decomposed iodides. The contribution concludes with a table in which the results obtained by this method closely correspond with those required by theory. C. A. M.

Titration of Thiosulphate Solution with Iodic Acid. E. Riegler. (*Zeit. anal. Chem.*, 1896, xxxv., 308.)—The reaction on which this is based is

$$6Na_2S_2O_3 + 6HIO_3 = 3Na_2S_4O_6 + 5NaIO_3 + NaI + 3H_2O.$$

For the indicator starch solution is added to the thiosulphate solution. As soon as the reaction is complete, the slightest excess of iodic acid sets free iodine, which is shown by the blue colour given to the starch.

$$5NaI + 6HIO_3 = 5NaIO_3 + 3H_2O + 6I.$$

Iodic acid is readily obtained in a state of purity; the crystals are not hygroscopic, and its aqueous solutions do not readily undergo change. In preparing the solutions for standardizing thiosulphate solution the pure crystallized acid is first dried over sulphuric acid. C. A. M.

LEGAL.

IMPORTANT DECISION WITH REFERENCE TO BEESWAX.

QUEEN'S BENCH DIVISION.

(*Before* MR. JUSTICE GRANTHAM *and* MR. JUSTICE WRIGHT.)

FOWLE *v.* FOWLE.

(*Reprinted from the "Pharmaceutical Journal" of November 7, 1896.*)

IN the Queen's Bench Division on November 2, before Mr. Justice Grantham and Mr. Justice Wright, the case of *Fowle* v. *Fowle* came before the Divisional Court, on a special case stated by the magistrates sitting at Cranbrook, in Kent, the appellant being the Inspector under the Sale of Food and Drugs Act, and the respondent a grocer at Marden. Mr. T. Mathew appeared for the appellant, but the respondent was not represented. The facts, as stated in the special case, were as follows: A man named Skinner, sent by the Inspector, went into respondent's shop and asked for a quarter of a pound of beeswax, which the respondent thereupon supplied.

Skinner asked if it was pure, and respondent said he could not guarantee it. Skinner said, "But you sell it as beeswax?" and the reply was, "Yes." Skinner thereupon took it away, and on being analysed it was found to contain 50 per cent. of paraffin. Definitions from Chambers' "Etymological Dictionary," and "The Lexicon of Applied Science," went to show that a drug was anything used in medicine, or any medicinal substance either used in its natural condition or prepared by art, or mixed with other substances to form a medicament or medicine; and the British Pharmacopœia was referred to as showing that yellow wax was used in the preparation of certain medicines, no less than twenty being mentioned, especially phosphorus pills. It was admitted that to obtain the wax mentioned in the British Pharmacopœia beeswax was melted, and certain impurities taken out. It was also said that the properties of this prepared wax, when used with oil, were of a healing and emollient character, and that it was used by chemists and druggists. A witness, who was a chemist (the name not being mentioned), was unable to say what was the medicinal or healing effect of beeswax alone, nor had he ever known it used alone. He described it as being used as a vehicle only, but in his opinion there was no manner of doubt as to its being a drug. On these facts it was contended by the appellant that it was a drug and by the respondent that it was not, and in any case that it was not the sale of a drug to the prejudice of the purchaser. The justices found: 1. That prepared beeswax, known as yellow wax, with the allied preparation known as white wax, is a substance mentioned in the British Pharmacopœia, and used in the preparation of medicines. 2. That wax used in the preparation of medicines is used as a vehicle only, and not as possessing any healing or medicinal properties, and that the sale of beeswax in this case was not in their opinion to the prejudice of the purchaser, because of the statement made by the respondent to Skinner. 3. That the sale of the said beeswax was a sale by the respondent as a grocer. On these facts they held that the beeswax in this case was not a drug within the meaning of the Act; and, further, that if it were, the said sale was not to the prejudice of the purchaser, nor was the sale by the respondent as a grocer a sale of a drug within the meaning of the Act.

Mr. JUSTICE GRANTHAM said there was no suggestion of any fraud here. The respondent kept a grocer's shop in a small place, and sold a variety of things, amongst them this beeswax. He did not know whether it was pure or not, and said so; it was probably a cheaper article than some of the finer qualities. He got that which was generally used, the constituents of which were paraffin and beeswax, which was probably more useful for some purposes than if it were pure wax. There was nothing improper in it, and he said he could not guarantee its being pure. It was very hard to say that the man should be punished because it turned out there was paraffin in it.

Mr. MATHEW suggested that the grocer would know that beeswax was a thing commonly used in medicinal preparations.

Mr. JUSTICE GRANTHAM thought it was about the last thing he would know about it.

Mr. MATHEW submitted that, though he might be allowed to sell it with its natural impurities, he could not be entitled to add to it a foreign substance which might be deleterious when used in medicine.

Mr. JUSTICE GRANTHAM said there was no suggestion that he added the paraffin himself; he had not the slightest doubt that he bought it as it was.

Mr. MATHEW went on to contend at some length that the beeswax in question must be considered the same as the yellow wax mentioned in the Pharmacopœia, which was used in making various medicinal preparations, and to point out the evil consequence which might ensue if a chemist purchased any of this wax from the respondent for use in the preparation, say, of phosphorus pills.

Mr. JUSTICE GRANTHAM said such a chemist ought to be fined very heavily for buying any drug from a small grocer in a little country town.

Mr. MATHEW suggested there would be no negligence if he believed it to be the proper article.

Mr. JUSTICE GRANTHAM said it would be worse than negligence; it would be guilty knowledge. A chemist ought not to buy drugs from a grocer.

Mr. MATHEW, continuing, said the grocer ought to tell the purchaser that it contained paraffin.

Mr. JUSTICE GRANTHAM said the grocer did not know what it contained.

Mr. MATHEW suggested that, if the justices were right in this case, it would be perfectly possible for a chemist dealing with yellow wax to adulterate it and sell it in the same way.

Mr. JUSTICE GRANTHAM said when such a case came before the Court they would deal with it.

Mr. MATHEW continued for some time longer to urge that beeswax was the same as yellow wax, and was a drug as being used in medicine, and cited several other dictionaries and cyclopædias.

Mr. JUSTICE WRIGHT asked whether bread, which was often used medicinally in the form of a poultice, was a drug.

Mr. MATHEW said he should contend, if it were mentioned in the Pharmacopœia, that it would be.

Mr. JUSTICE GRANTHAM suggested that Quaker oats would also come within the definition, and also water, which entered into most fluid mixtures.

Mr. JUSTICE WRIGHT also put the case of wine and brandy.

Mr. MATHEW said they were clearly not drugs, but anyone who adulterated wine would be liable under the term "food or drink." He continued the argument for some time longer, and cited various cases, one of which was with regard to the sale of saffron.

Mr. JUSTICE GRANTHAM, in giving judgment, said as no one appeared on behalf of the respondent in this case, the Court had not had the benefit of the argument which would have been used by counsel on his side, but it was the duty of the Court to watch carefully the interest of a person not represented, and make such suggestions as occurred to them, whether for or against the argument presented by the appellant. It certainly struck both his learned brother and himself that there were a great many difficulties in his way, and they had come to the conclusion that it was not desirable to interfere with the decision of the magistrates in this case, but that it was the right decision. In the first place, speaking for himself, he was not prepared to admit that beeswax was a drug when sold as it was by a grocer in a country shop, and not sold as a drug by him. It might be possible by straining a point, and going into all the pharmacopœias and dictionaries that could be found, that in one or more of them you might find such a definition as would bring this within it, but it was quite clear from most of the definitions read by the learned counsel that this would not come within it, although it might in some others. He thought the magistrates were right in referring to the fact that this was sold by a grocer; everyone could no doubt think of instances in which beeswax was used not as a drug. The primary use of beeswax used to be to polish mahogany tables, and you might use a drug for that purpose; but that was not a medicinal purpose, except so far as it would act medicinally for the benefit of the person polishing the table using a great deal of elbow grease, which would be a very healthful occupation. Not to give other instances in which beeswax was used which were certainly not medicinal, he should think not one grocer in a thousand ever used beeswax as a drug; he sold it for the ordinary purposes of trade. As he had said before, if a doctor were to buy beeswax as a medicine he would not go to the ordinary country grocer and buy the cheap beeswax which was sold there; it would be very culpable and negligent on his part if he did. The magistrates had to deal with the facts as they were and people as they found them, and the knowledge they had of the kind of trade they did. It seemed to have been assumed, on behalf of the appellant, that the grocer made his beeswax, but that was not

so; he bought it. He was asked if it was pure; he said, "I do not know; I cannot tell you that it is pure; it is what we sell; we have to sell beeswax; but I do not know what its constituents are." Thereupon the man bought it, and it would be very hard indeed after that if he were to come and say, because there was some other substance in it, he was to be fined. Under these circumstances he thought the magistrates were quite right in dismissing the application.

Mr. JUSTICE WRIGHT said he was of the same opinion, on the ground that it must be in many cases a question of fact whether a particular thing was a food or a drug or something else, which the magistrates must determine in each case. In this case he thought they had rightly determined. Take, for instance, turpentine. Turpentine, if sold to be used by a carpenter, ought not to be held to be a drug for the purpose of this Act, or resin. He supposed both turpentine and resin were used in medicine, and if sold by a chemist to be used in medicine it would be a drug; but if resin were sold to be used by a musician he should say it was not a drug. Another point which seemed to him much more important, whether the mere fact of the seller saying that he could not guarantee the article to be pure would prevent it being to the prejudice of the purchaser, he would rather not decide at present; but certainly the case cited was a very different one from the present.

REVIEW.

APPLIED BACTERIOLOGY. By T. H. PEARMAIN and C. G. MOOR. (London: Baillière, Tindall and Cox.) Price 12s. 6d. net.

This work, which is primarily intended as a handbook for students and medical practitioners, contains much that will be found of great practical value to the analyst, who is now so frequently called upon to undertake bacteriological investigations. In order to keep the book within the limits of a handy size, the authors have endeavoured to select from the vast mass of material at their disposal only those portions the results of which have been established, or are likely to be so in the near future. The practical portion of the work, as might be expected from the wide experience of the authors, has been exceptionally well done. In the chapter referring to the "Methods of the spread of infection," the subject has been treated in a somewhat novel and ingenious manner. An elaborate description is given of the bacterial organisms which are the cause of the more commonly occurring infectious diseases of man, with the special methods for their individual identification. This is followed by a brief description of the principal yeasts and mould-fungi, and of the more commonly-occurring enzymes, together with their fermentative actions. Next comes a chapter on ptomaines and on the biological testing of disinfectants, followed by another on the examination of water, filters, air, and soil, to which is appended a succinct description of a number of micro-organisms not mentioned in the preceding pages. Steam sterilizers are separately treated of in the appendix. The work is well printed and illustrated, and especial reference must be made to the beautifully-executed colored plates representing the bacteria and the manner of their growth when cultivated under different conditions. We congratulate the authors on the production of a really useful book, eminently adapted for the wants of those who, like analysts, only require the essence of the subject given in a lucid and terse form. W. J. S.

THE ANALYST.

FEBRUARY, 1897.

PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS.

THE annual general meeting was held on Wednesday, January 13, 1897, in the Chemical Society's Rooms, Burlington House, the President (Dr. Stevenson) occupying the chair.

The minutes of the previous meeting were read and confirmed.

In the absence, through illness, of the Hon. Treasurer (Mr. E. W. Voelcker), his report and the accounts for the past year were presented by Dr. J. Augustus Voelcker, and, on the proposal of Mr. Archbutt, seconded by Mr. Ekins, were unanimously adopted.

Mr. Archbutt and Mr. Budden were appointed to act as scrutators of the ballot-papers.

It was proposed by Mr. Smetham, and seconded by Mr. Page, that a vote of thanks should be accorded to the Hon. Treasurer and Hon. Auditors for their services during the past year. The motion was carried unanimously.

The following gentlemen were nominated by the Council for election as Associates: Mr. Arthur Angell, jun., assistant to Mr. Arthur Angell, Southampton; Mr. W. H. Jackson, assistant to Mr. A. C. Wilson, Stockton-on-Tees; and Mr. Arnold Rowsby Tankard, assistant to Mr. Alfred H. Allen.

Mr. Frank H. Stephens was elected a member.

The PRESIDENT then delivered the following address:

THE PRESIDENT'S ANNUAL ADDRESS.

The past year has been one marked in the annals of the Society by quiet, steady progress, and some important events to which I shall presently refer in detail.

During the year we have elected 1 Honorary Member and 11 new ordinary Members, also 6 Associates, and 1 Associate has been transferred from the list of Associates to that of Members. On the other hand, 1 Member and 1 Associate have resigned, and the names of 2 Members and of 1 Associate have been removed from the list for non-payment of subscriptions. The respective numbers of the Members and Associates as compared with those of the previous year are therefore as follows:

	January, 1896.	January, 1897.
Hon. members	10	11
Members	210	218
Associates	26	29
Total	246	258

The following papers were presented to the Society:

"Note on a series of Analyses of a Private Water Supply." By E. Russell Budden.
"The Determination of Oxygen in Commercial Copper." By Bertram Blount.
"The Composition of Milk and Milk Products." By H. Droop Richmond.
"A new form of Carbonic Acid Apparatus." By Cecil H. Cribb, B.Sc.
"Laboratory Notes." By Alfred H. Allen.
"On the Estimation of the Diastatic Power of Malt." By Walter J. Sykes, M.D., and C. A. Mitchell, B.A.
"Further Note on the Detection of Formalin." By H. Droop Richmond and L. K. Boseley.
"The Detection of Formalin." By Otto Hehner.
"Note on the Detection of Cotton-seed Oil in Lard." By E. J. Bevan.
"The Bacteriological Examination of Water for the Typhoid Bacillus." By T. H. Pearmain and C. G. Moor, M.A.
"Note on the Estimation of Formic Aldehyde." By Harry M. Smith.
"Note on Hehner's Test for Formic Aldehyde." By Norman Leonard, B.Sc.
"The Composition of Human Fat." By C. A. Mitchell, B.A.
"Note on an Incrustation found in Hot-water Pipes." By J. Augustus Voelcker, M.A., B.Sc., Ph.D.
"The Examination of Commercial Milk Sugar." By H. Droop Richmond.
"Note on 'drawn' or exhausted Caraways." By Bernard Dyer, D.Sc., and J. F. H. Gilbard.
"The Analysis of Mortar." By W. J. Dibdin and R. Grimwood.
"Commercial Prussian Blue." By Ernest J. Parry, B.Sc., and John Henry Coste.
"Commercial Cream of Tartar." By Alfred H. Allen.
"Note on Lead in a Sample of Canadian Cheese." By F. Wallis Stoddart.
"Note on Ginger." By Thos. P. Blunt.
"The Determination of Stearic Acid in Fats." By Otto Hehner and C. A. Mitchell, B.A.
"Further Note on Lead in Canadian Cheese." By F. Wallis Stoddart.
"Note on Formalin." By Samuel Rideal, D.Sc., and Ronald Orchard.
"On the Separation and Estimation of Formic Aldehyde." By Norman Leonard, B.Sc., and Harry M. Smith.
"Notes on Prussian Blue." By Frank H. Leeds.
"The Estimation of Borax and Boracic Acid in Milk." By Frank P. Perkins.
"Note on Copper in Oysters." By W. F. Lowe.
"The Statement of Analytical Results." By J. F. Liverseege.

The papers read before the Society—twenty-nine in number—have as a rule not presented any such marked characteristics as to render them landmarks of great advances, with the exception of that on "The Determination of Stearic Acid in Fats" by Messrs. Hehner and Mitchell. This paper, indeed, is eminently suggestive, and

promises an entire reconstruction of our existing and too imperfect methods of analysing fats.

I trust that our members appreciate the improved and extended form in which our organ, the ANALYST, has appeared during the year that has just passed. Under the able editorship of Dr. Sykes, and under the direction of the Publishing Committee —where the attendance has been regular and good, and the criticisms and discussions careful and valuable—the ANALYST has made a distinct advance. It is now the only journal for analysts appearing in this country worthy of the name. In it, besides the original articles above named, there have appeared in abstract the following number of papers:

Food and Drug Analysis	55
Toxicological Analysis	9
Organic Analysis	91
Inorganic Analysis	86
Apparatus	9
	250

In the early months of the year 1896 the judgment in a Divisional Court, Queen's Bench Division, of Hawkins J. and Kennedy J., in the case of Fortune *v.* Hanson, as to the insufficiency of a certificate relative to milk, engaged the attention of your Council. After mature deliberation and with excellent legal help, a circular was issued on April 11 last expressing the views of the Council as to the forms of certificate that should be used in the cases of watered milk and watered butter respectively. Some magistrates refused to accept our amended form of certificate relative to milk. But a Divisional Court of the Queen's Bench Division, by the judgment of Grantham J. and Kennedy J. in the case of Bridge *v.* Howard, affirmed the sufficiency of our form of certificate, which will now I hope be generally adopted as explicit, safe, and sufficient. This judgment is the more satisfactory as one of the two judges giving it was also one of the judges who decided the previous case of Fortune *v.* Hanson.

By far the most momentous event of the year has been the issue of the Report of the Food Products Adulteration Committee, a committee which, fortunately, was able to bring to a close its protracted sittings in time for its report to be presented to the House of Commons before the prorogation of Parliament. Our distinguished Past President, Mr. Hehner—and, I may add, on the special request of your President. and I am sure with your approval—most ably and vigorously gave clear, emphatic, and impartial evidence before the committee, and obviously deeply impressed it. Indeed, he proved himself as skilful a Field-officer as we had previously known him to be a General of consummate tact and ability. Again, in the name of the Society, let me tender him our warmest thanks for all that he has done to advance the views of the Society and the aims of our best public analysts. In my further remarks I have to acknowledge the assistance he has so generously afforded me in dissecting the recommendations of the committee.

The report of the committee has two very distinct aspects. If the "Summary of Principal Recommendations," set out on page 41 of the report, is alone read by anyone not having the patience to wade through the whole report, the impression

gained would be unfavourable to the views of our Society, and an insufficient and incorrect view would be obtained of the gist of the report, for there is a strange discrepancy between the report itself and the summary of its principal recommendations. Indeed, in the summary many of the things of real importance recommended by the committee in the body of the report are either so inadequately stated or omitted that, if the summary alone is looked at, it would appear that the committee desired to make the proposed Act worse from the public analysts' point of view than the existing Act. A careful perusal of the body of the report will, however, convince the reader that the report is written in a much juster spirit than would appear from the summary. In the first place, the committee state that the existing Acts, where well administered, have had most beneficial results, and that the better they have been worked the more adulteration has declined. In accordance with our draft Bill, the committee emphasise the necessity of having at least one sample analysed annually per 1,000 inhabitants of a district; but the committee were evidently not familiar with the Sale of Food and Drugs Acts, for they recommended that the County Councils should force the local authorities to carry out the Acts, apparently forgetful of this—that the County Council is itself the local authority, and has no jurisdiction, *quâ* the Acts in question, to compel local authorities to procure samples and have them analysed.

As to the giving of notice by the vendor to a purchaser that an article is mixed, it is recommended that for a verbal notice an obligatory and legible labelling of the mixture should be adopted. This is in accordance with our recommendation, except that the report does not go the length of advising that the proportions of the respective ingredients of a mixture, or even that the nature of the components, be required to be stated. Doubtless it was thought by the committee that it was not desirable to enforce the disclosure of trade secrets, and that an attempt to do this would be inoperative.

The recommendation as to warranties is practically that advised by our Society, though, if the summary only be consulted, an inadequate impression of the far-reaching character of the intended use of warranties would be obtained. Invoices are to have the force of warranties so far as the retailer is concerned; but only on conditions—the incidence of the prosecution is to be transferred to the wholesale dealer.

As to the examination of imports by the Customs, the intention of the committee is good; but as the committee is against the stoppage in transit of articles found adulterated, the proposed examination by the Customs will probably be without result. The inspectors ought to be empowered to take the article without delay to the local public analyst, and should be empowered to take proceedings on his certificate, instead of incurring the delay involved in the reference to a central public laboratory.

Importers are to submit guarantees of the purity of their food imports to the scrutiny of the Customs; these guarantees to be given by the foreign vendors, together with evidence that the importers have taken proper measures to see that the goods are such as they are guaranteed to be. The excuse of ignorance will thus be removed.

And in this connection I may refer with satisfaction to the recent conviction of a wholesale firm of English dealers in lard, for the sale, under a false name, of lard

containing 10 per cent. of beef-stearin, but guaranteed as pure refined lard. This conviction has not been appealed against, and I hope it will greatly tend to the stoppage of the sale of such articles under a misleading title. No imputation, except of carelessness, was made against the wholesale dealer, who was not the manufacturer of the lard; nevertheless he was convicted.

The recommendation, in accordance with the expressed views of this Society, that the present provision as to samples of milk being taken for analysis in transit should be extended to all articles is satisfactory.

The contemplated provision for a purchased sample being divided into four parts, one of which is to be at the disposal of the wholesale dealer, is theoretically a most just and equitable recommendation. It, however, if adopted, will greatly increase the difficulties of administration. Many articles are costly, and the purchase of unusually large quantities in order to have sufficient for four independent analyses, will excite suspicion as to the intention of the purchaser. In the case of tinned articles, the further recommendation that a retailer shall be empowered to refuse to sell an article otherwise than in a manufacturer's unopened labelled tin or packet, increases the difficulty. No exception can be taken to the recommendations that all certificates, whether of a public or Government analyst, must be given within twenty-eight days, and that magistrates may not refuse a reference analysis to Somerset House.

But the recommendation that the certificate of a public analyst employed by the defence should be received on equal terms with that of the certifying public analyst whose certificate is in question is a very doubtful proposed innovation. Happily, the certificate of anyone not a public analyst is not admissible, and the care to be exercised in the determination of the qualifications of a public analyst to be hereafter demanded, remove some of the objections to the departure from existing use. It will, however, behove a public analyst, when receiving unknown samples, to take care that he is not imposed upon to the detriment of one of his official professional brethren, and it will be incumbent on our Society to strictly watch the conduct of public analysts in this regard.

In one respect the report goes beyond the recommendations of this Society, made in its Draft Bill. Penalties for adulteration are to be increased. It will be remembered that the reason why this Society did not make this recommendation was that penalties were considered to be outside the province of public analysts.

Very important and welcome are the proposals as to a more stringent and specific test of the qualifications of a candidate for the post of public analyst being prescribed, coupled with the statement of the committee that hitherto the duties of the office have usually been discharged with commendable efficiency, and the advice that remuneration by way of both stipend and fees is best calculated to secure the services of the most competent analysts. It is also proposed to make his remuneration subject to the approval of the Local Government Board.

The proposed extension of the definition of the word "food" as used in the Acts, so as to make it include expressly all articles intended to enter into or be used in the preparation or flavouring of food, is a welcome extension of the law; and it is to be hoped that if adopted by the Legislature it will put an end to the sale of presumptively noxious, though not actually poisonous, baking powders and the like articles.

But this leads up to the most important of all parts of the report—the adoption by the committee of the principle of the establishment of a Scientific Court of Reference under the Board of Trade, and independent of the Government Laboratory. This recommendation, and the admission of the necessity for it, met with much opposition; but the able advocacy of Mr. Hehner was irresistible. The words of the committee are clear and emphatic as to the necessity for such a reference tribunal. After referring to the absence of food-standards, or precise definitions of the composition of articles of food to which the Act is understood as referring, and which the purchaser is presumed to have in mind when making a purchase, they say that the article demanded must be supposed to mean an article of the nature, substance, and quality of the article usually sold under that name, and as to the variation of the composition of many articles of food sold under one and the same name, they add that there is a substantial reason for dissatisfaction with the existing provisions of the law upon this subject, and the decisions by tribunals seldom in possession of sufficient data for the satisfactory decision of such questions. Hence they point out how the working of the law as to the adulteration of food would be facilitated, and the interests of traders served, if standards or definitions of foods were promulgated by competent authority. This cardinal principle, for many years advocated by this Society, has thus received its due recognition, and there can be no doubt that when a Bill is introduced by the Government this provision for a Court of Reference must form part of its provisions, and, in the words of the committee, the Reference Tribunal must be a specially constituted scientific body, familiar with questions of analysis and the chemistry of food. All this I heartily endorse.

But as to the Court of Reference, the committee do not give us in their recommendations all that we asked for, or all that justice demands. We are not to be directly represented in that body. Who are so familiar with foods, their composition and analysis, as the members of our own body, which embraces practising analysts of the highest distinction in every department of food and drug analysis? What body of men in this empire has more advanced scientific analysis than our Society? What British Journal more teems with original analytical research than ours? The Chemical Society has practically long ceased to take interest in analytical work, except as to the means of determining formulæ; the Pharmaceutical Society only advances analysis in a limited field; and the Institute of Chemistry is an examining and qualifying body only. The Society of Public Analysts has now for a quarter of a century been the pioneer of analytical progress in this country. The result of the committee's deliberations is a compromise. An unworthy proposal to exclude public analysts altogether from the Court of Reference was, however, happily rectified. There is no doubt that public analysts will sit in the Court of Reference, which would be an incomplete and incompetent court without them. The Institute of Chemistry and its Council contain so many able and prominent chemists who are also public analysts, that there is little doubt that the apparent unfairness of the exclusion of public analysts from a seat on the proposed Board will be rectified unless, by some strange fatality, the members of the Institute of Chemistry, which is to be represented, be also excluded from the provisions of the forthcoming Bill. But that contingency is not likely to occur. For my part, I

think that it is immaterial how the members of the court are selected, provided that competent persons be secured whose decisions will command respect. Did I think the "analysts of repute," among whom the representative of the Institute of Chemistry finds a place, would not include public analysts, I, for my part, would vigorously protest against the finding of the committee; but I think we may congratulate ourselves on having gained, though indirectly, all that we demanded as to a Court of Reference.

It is a matter for satisfaction also that the unwillingness of the officers of the Government Laboratory to communicate information to the Society of Public Analysts, lest we should think such action to be dictation, is gently, but firmly rebuked, when the committee state that they regard it as of great importance, in the interests of the public, that public analysts should, as far as possible, be made acquainted with the methods adopted by the Government Laboratory in the analysis of food, and with the considerations kept in view by them in determining whether an article has been adulterated. Doubtless we may expect this emphatic pronouncement to have a generous and not tardy response, and the official book, which ought to be, and possibly is, in course of preparation by Somerset House, placing all the analytical methods used in food and drug analysis, and the deductions that ought to be drawn from the results of analysis at our disposal, will be welcomed by public analysts as a priceless boon.

The report of the committee may be considered as, on the whole, a recognition of the principles advocated by this Society, a testimony to our usefulness, and of the ability and discretion with which public analysts have discharged their duties, of the fewness of the errors committed by us, and an admonition to the Government analysts to be less grudging of information than of yore. Nearly every vital point advocated by us has been gained.

The committee, indeed, in its report and recommendations, though of course it has not endorsed all the views put forth by the Society in its Draft Bill so well known to us—has substantially adopted most of our recommendations.

That portion of the report which deals with specific articles, such as coffee and cocoa, is of less importance, seeing that the Board of Reference will manifestly have to determine the composition of all such articles and their legitimate mixtures.

The invaluable aid rendered to the Society by Mr. Kearley, M.P., with reference to contemplated new legislation, received the only recognition which this Society could bestow, by his being unanimously elected an honorary member of the Society.

There can be little doubt that the Government ought to feel it incumbent on them to introduce into Parliament a Bill, based on the report of the committee, at an early date, and it is devoutly to be wished that the Bill may not be crowded out by a plethora of less important matters. Traders, public authorities, and public analysts are all desirous that a Bill may not be delayed, and that it may speedily be enrolled in the Statute Book.

Let me congratulate the Society on what it has done to forward these matters.

Mr. CASSAL said that he had great pleasure in rising to propose that the Society should tender to Dr. Stevenson its cordial thanks for his valuable services as

President during the past year, and for the admirable address to which they had just listened. He was sure that all who had had the opportunity of sitting under Dr. Stevenson's guidance at the Council-board of the Society, and all who had been in the habit of attending the meetings so genially presided over by him, would agree that Dr. Stevenson had been essentially the right man in the right place. He had been an admirable leader of the Society and of the profession during two momentous years, perhaps the two most momentous years in the existence of the Society since the passing of the Sale of Food and Drugs Act in 1875. He (Mr. Cassal) ventured to think that Dr. Stevenson had worthily filled the chair, and had ably supported a policy which, without being charged with invidiousness, the speaker thought he might say had been initiated by Mr. Hehner when President of the Society. He (Mr. Cassal) would have liked to enlarge at greater length upon all that the Society owed to Dr. Stevenson; but the annual dinner was looming in the very immediate future, and he would therefore now ask the meeting to express the Society's thanks to Dr. Stevenson for the services which he had rendered to it, and to wish him in his retirement all prosperity and happiness.

Mr. W. F. Keating Stock had much pleasure in seconding the motion, which, on being put to the meeting by Mr. Cassal, was carried unanimously amidst loud applause.

Dr. Stevenson said he desired to thank the meeting heartily for the vote which had been passed to him in such a cordial manner. It had been a very great pleasure to him to preside over the Society. He was glad to think that he had been able to conduct the affairs of the Society amicably during his term of office, and, he trusted, successfully. He was glad to be able in his address to congratulate the Society upon its success in securing the report which had been made by the Select Committee on Food Products Adulteration, and the carrying out of the policy which, as Mr. Cassal had justly said, was so greatly contributed to by Mr. Hehner. In laying down the cares of office and going into retirement, he did not intend, as Mr. Cassal seemed to intimate, to absent himself in future from the meetings. His attendance might not perhaps be so constant as it had been during the past two years, but he hoped to be with the Society very frequently, and to retain his interest and influence in its proceedings.

The following papers were read:

"A Specific Gravity Pipette." By W. F. Keating Stock.

"Note on Weighing out Fats." By Charles E. Cassal.

"A Modified Schmidt Process." By R. W. Woosnam.

The scrutators reported that the list of Officers and Council elected for 1897 was as follows:

President.—Bernard Dyer, D.Sc.

Vice-Presidents (who have filled the office of President).—M. A. Adams, F.R.C.S.; A. H. Allen; Sir Chas. A. Cameron, M.D., F.R.C.S.; A. Dupré, Ph.D., F.R.S.; Otto Hehner; Alfred Hill, M.D., F.R.S.E.; J. Muter, M.A., Ph.D., F.R.S.E.; Thos. Stevenson, M.D., F.R.C.P. (Who have not filled the office of President)—W. P. Aitken, D.Sc., F.R.S.E.; W. W. Fisher, M.A.; John Pattinson.

Hon. Treasurer.—E. W. Voelcker, A.R.S.M.

Hon. Secretaries.—E. J. Bevan, Charles E. Cassal.

Other Members of Council.—Leonard Archbutt, Bertram Blount, A. Wynter Blyth, M.R.C.S., R. Bodmer, E. Russell Budden, A. C. Chapman, W. J. Dibdin, Sidney Harvey, S. Rideal, D.Sc., Alfred Smetham, J. E. Stead, John A. Voelcker, M.A., B.Sc., Ph.D.

The annual dinner subsequently took place at the Criterion Restaurant.

THE USE OF ANTISEPTICS IN FOOD.

(*Abstract of Report of the "Lancet" Special Commission.*—"*Lancet*," 1897; p. 56.)

THE Select Committee appointed by the House of Commons on Food Products Adulteration did not offer a pronouncement on the question of antiseptics in food, but expressed themselves as follows: "Your committee think that the matter is one which deserves further investigation by recognised scientific authorities, with the view to the expression of an opinion that would be regarded as authoritative." In another section of their report they mentioned the legitimacy of the use of preservatives as a subject for consideration by the proposed Court of Reference.

It being obvious that an authoritative expression of opinion must rest on the evidence and experience of medical men of recognised authority, the Editors of the *Lancet* addressed a letter to several leading members of the profession, containing the following questions:

(1) "Is the presence of small quantities of salicylic, boric, or benzoic acids, or 'formalin' in food, in sufficient quantities to preserve it, injurious to health?"

(2) "Should the use of antiseptics for this purpose be forbidden by law altogether?"

(3) "Should legislation be brought to bear on the restriction of the amount?"

(4) "Should the law insist that when preservatives are used the fact should be stated on the label?"

In answer:

Sir HENRY THOMPSON wrote that "he had long held that the addition of antiseptics was undesirable, though unable to produce evidence that any one of them had given rise to deleterious action, owing to the impossibility of isolating the precise influence of the drug. He objects strongly to the *dietetic* use of drugs, and is of opinion that the name and quantity of the antiseptic employed should be on the label, or on a paper setting forth the maker's or vendor's name."

Dr. PAVY wrote that "he did not consider our knowledge sufficiently extended to permit of its being taken for granted that no injury is producible, though there is no evidence of injury to health. He points out that it is the vendor, and not the consumer, that is benefited. He considers that notification of the fact of antiseptics being employed, and their nature and amount, would be sufficient; any deviation from the notification should be liable to prosecution. With the public interest thus

safeguarded, he thinks that advantage might be taken of the power of antiseptics in preserving articles of food."

Dr. F. J. ALLEN points out the possibility of daily accumulation of antiseptics quite sufficient to produce a gradual lowering of the standard of health, and is of opinion that the fact of an antiseptic being added, and its nature, should be required by law to be announced at the time of sale.

Dr. SIMS WOODHEAD draws attention to idiosyncrasy and cumulative effect, and dwells upon our ignorance of the action of certain drugs (*e.g.*, formalin) on food stuffs. He points out that by the use of preservatives foods of inferior quality may be "doctored." He would make the use of antiseptics illegal unless their nature and quantity be made known.

The late Sir B. W. RICHARDSON considered that antiseptics are not only necessary at this moment, but when used in proper form and quantity, cause no injury whatever. There ought to be a license given permitting a certain fixed, and not a dangerous, quantity of antiseptic, and it ought to be stated on the label what the antiseptic is and its quantity.

Dr. T. LAUDER BRUNTON writes that "one must remember that poisons are formed in foods by spontaneous decomposition, which may take place after purchase. The question to be decided comes to be whether antiseptics are likely to be more injurious to health than the natural products of decomposition. His own belief is that preservatives are the less injurious. His answers are: (1) The use of antiseptics should not be forbidden by law. (2) It is doubtful whether legislation should restrict the amount, as the makers will probably use the minimum amount found sufficient. (3) The fact of preservatives being used, and their amount, should be stated on the label."

Sir W. ROBERTS says that "there is no reliable information available, and an inquiry is needed."

Dr. W. D. HALLIBURTON is not able to give information as to injurious effects from his experience, but quotes F. J. Allen as mentioning cases of ill-health in children due to boric acid.

Dr. J. B. BRADBURY thinks that "it is not necessary to forbid antiseptics, but that the amount should either be restricted, or the fact of their addition stated on the label."

Dr. WHITELEGGE cannot speak positively, though it is clear to him that the law should insist upon a plain statement on the label if any preservative be added.

H. D. R.

ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

FOOD AND DRUGS ANALYSIS.

Modifications in the Composition of Brandies through Age. X. Rocques. (*Ann. de Chimie Analyt.*, vol. i. [20], pp. 385-389.)—The conclusions deduced by the author from his researches are to the effect that the co-efficient of impurities—volatile substances other than ethyl alcohol—increases considerably with age; the proportion

of oxidation products (acids and aldehydes) is also largely increased; the proportionate increase of the higher alcohols and ethers is much smaller, and only relative, being chiefly due to concentration; the ratio of the higher alcohols to the ethers is larger; and the total quantity of volatile substances, apart from ethyl alcohol, is about the same in *corresponding* volumes of new and matured brandy, the gain in oxidation products compensating the loss in ethers and higher alcohols. C. S.

Estimation of Salicylic Acid, and its Detection in Wine, Beer, etc. F. Freyer. (*Chem. Zeit.*, 1896, xx., 820.)—This paper consists chiefly of a description of the tribromophenol method of determining salicylic acid; and, provided that at least twice as much of the bromine is present as is required in the reaction, the author finds that it is possible to obtain satisfactory results, except in the case of wine and beer (as has already been pointed out by Elion).

The process for the detection of salicylic acid in the latter liquids is based on its volatility in presence of steam, and its colour reaction with ferric chloride. Fifty or 100 c.c. of the sample are distilled until two-thirds have passed over; the receiver is changed and the process continued till only 5 c.c. are left in the retort. A trace of ferric chloride is then added to the second distillate, preferably by stirring it with a rod moistened with a dilute solution, and any red coloration noted. The method is available if the wine contain not less than 0·02 gramme of salicylic acid per litre; it avoids the tedious extraction with ether, etc. (*cf.* ANALYST, xx., 184), and it has the additional advantage of enabling the alcohol in the liquid to be also determined at one operation (in the first distillate). Should a smaller percentage of the preservative be suspected, the ordinary treatment with petroleum ether must be carried out, as this will detect 0·01 gramme per litre (*cf.* Spicer, *loc. cit.*).

The author has been unable to check the process on those wines that, as Medicus has observed, naturally give a petroleum ether extract coloured violet with ferric chloride; but as this reaction is only obtained when at least 100 c.c. are used, perhaps not more than 50 c.c. of the sample should be taken in testing for salicylic acid.

F. H. L.

The Effect of Coal-Tar Colours on the Process of Digestion. H. A. Weber. (*Amer. Chem. Jour.*, 1896, xviii., 1092-1096.)—Experiments on lower animals have led to the belief that most of the coal-tar colours used for confectionery purposes are quite harmless; but as there was no published account as to their influence on digestion, the author has tested some of those most commonly employed.

The ferments used were Armour's pepsin and pancreatin, and the substance to be digested, blood fibrin. In each experiment on peptic digestion a mixture composed of hydrochloric acid solution ($\frac{2}{10}$ per cent.), 100 c.c.; pepsin, 20 milligrammes; and fibrin, 1 gramme, was digested in the water-bath at 38°-40°, while in a similar series of tubes weighed quantities of the colour were added. Any fibrin remaining undissolved after the time required to dissolve that in the control tube was removed, washed, pressed between filter-paper, and weighed.

Pepsin and Oroline Yellow.—This colour, known in the trade as *acid yellow* or *fast yellow*, is a mixture of sodium amido-azo-benzene-di-sulphonate with sodium amido-azo-benzene-mono-sulphonate. The experiments show that it has a marked and injurious effect on peptic digestion. In each case the amount of fibrin taken was 1 gramme, that of the pepsin 0·020 gramme, and the duration of the digestion was three hours.

	Colour. grm.	Fibrin dissolved. grm.		Colour. grm.	Fibrin dissolved. grm.
1.	0·0	1·0	4.	0·25	0·22
2.	1·0	0·1	5.	0·125	0·35
3.	0·5	0·12	6.	0·062	0·73

The experiments were also carried out with a $\frac{6}{10}$ per cent. solution of hydrochloric acid, with similar results.

Pepsin and Saffoline.—This colour, which was found to be *acridine red*, slightly retarded the digestion of the fibrin, but on the whole the author considered its effect to be practically *nil*.

Pepsin and Magenta.—The fibrin contained in the five test tubes with the same proportions of colour as before dissolved in every case in three and a half hours, so that magenta appears not to interfere with peptic digestion.

For the experiments on pancreatic digestion, the mixture consisted of: water, 100 c.c.; sodium bicarbonate, 1·5 grammes; pancreatin, 0·3 gramme; fibrin, 1·0 gramme. The quantities of pigment added to each tube and the method of digesting were the same as before.

Pancreatin and Oroline Yellow.—This colour, which had so marked an effect in retarding peptic digestion, was found to have no action whatever on the pancreatic ferment, the fibrin in every case dissolving as freely as in the blank experiment.

Pancreatin and Saffoline.—The duration of the experiment was six and a half hours, and the amounts of fibrin and pancreatin 1 gramme and 0·3 gramme respectively.

	Colour. grm.	Fibrin dissolved. grm.		Colour. grm.	Fibrin dissolved. grm.
1.	0·0	1·0	4.	0·25	0·55
2.	1·0	0·0	5.	0·125	0·65
3.	0·5	0·0	6.	0·062	0·75

Tannin precipitated the colouring matter.

Pancreatin and Magenta.—This had as marked an effect as saffoline in retarding digestion.

	Colour. grm.	Fibrin dissolved. grm.		Colour. grm.	Fibrin dissolved. grm.
1.	0·0	1·0	4.	0·25	0·40
2.	1·0	0·0	5.	0·125	0·60
3.	0·5	0·0	6.	0·062	0·73

The solutions in tests 2 and 3 gave no precipitate with tannin, but in all the others the precipitate was either marked or heavy.

Pancreatin and Methyl Orange.—This behaved precisely like the last two, completely stopping the digestion in the two stronger solutions, and retarding it in the three weaker.

From the examination of these four colours, it appears that while none interfere with both peptic and pancreatic digestion, all interfere with one or the other, and are therefore very undesirable in food or drink. C. A. M.

The Detection of Chestnut Leaves in Powdered Senna. L. E. Sayre. (*Amer. Jour. Pharm.*, 1896, lxviii., 587, 588.)—About 0·5 gramme of the No. 60 powder is placed in a layer of uniform thickness on a clean glass slide, below which is a sheet of white paper. One drop of a 5 per cent. solution of ferric chloride is allowed to fall upon the powder, and remains for some time, in the form of a globule, unabsorbed. If any chestnut leaves be present, the particles under the drop will turn a dark blue or black (tannin) in less than thirty seconds. It is stated that a very small percentage of the adulterant can thus be detected.

Under the microscope the adulteration may often be detected by the presence of the tracheids and pitted cells which compose the mid-rib of the chestnut leaf.

C. A. M.

On Maté or Paraguay Tea. B. A. Katz. (*Zeit. Nahr. Untersuch*, 1896, x., 364.)—Pure maté should consist only of the leaves of the *Ilex Paraguayensis*, which have a green colour and a characteristic aromatic smell. It appears, however, that much of that sold in Europe, especially when powdered, is mixed with the stalks of the plant, which contain no caffeine and are valueless. The author's investigations show that the ash from the extract of maté leaves contains a high percentage of manganese and magnesium salts, to which it is thought the plant probably owes some of its physiological properties.

The mean results of the analyses were:

	Per Cent.
Mineral matter	7·24
Moisture...	9·38
Total nitrogen	2·05
Caffeine	1·15
Albuminoids = (N, excluding that of caffeine, × 6·25) ...	10·75
Fat and resin	6·57
Tannin	7·74
Substances soluble in water	31·18
Per cent. ash soluble in water...	36·05
,, ,, ,, HCl	71·24

It is stated that for some time past the young leaves of *Ilex aquifolia* have been used in the Black Forest in the place of Chinese tea. C. A. M.

ORGANIC ANALYSIS.

Examination of Bergamot Oil. A. Borntraeger. (*Zeit. anal. Chem.*, 1896, xxxv., 523-525.)—In a former communication (ANALYST, xxi., 137) the author recommended the determination of the amount of linalol acetate as a test of the purity of an oil, basing his calculations on the standard of 38 per cent. In their recent report,

however, Schimmel and Co. state that for the last crop this amount was seldom reached. In ripe and semi-ripe fruits it was 33-34 per cent., rising to 37 per cent. when the fruits were ripe. Only in a few instances of Parthian bergamot oil towards the close of the season was 40-44 per cent. of linalol acetate found. The difference between these figures and those found by the author and Campolo is largely explained by the fact that the latter were obtained entirely from the examination of sound and ripe fruits of an earlier crop.

The author concludes, therefore, that it is necessary to supplement the saponification process by the density and optical rotation of the oil, and asserts that he never intended the determination of these constants to be superseded. C. A. M.

A Reaction for Morphine. C. Kippenberger. (*Zeit. anal. Chem.*, 1896, xxxv., 421.)—On adding to the solution of a morphine salt an alkaline hydroxide, and then a solution of iodine in potassium iodide, containing as little as possible of the latter salt, a yellow colour is obtained, which, on the gradual addition of more iodine, changes to a grass green. This is probably due to a process of oxidation. An analogous reaction is obtained with bromine, but is more uncertain than in the case of iodine. Chlorine by itself does not give the colour, but on adding potassium iodide to the solution after the addition of chlorine, it is immediately produced. Apomorphine, treated in the same manner, gives a brown coloration and then a precipitate. Other alkaloids, including codeine, do not show any abnormal behaviour with the reagent. C. A. M.

A New Method for the Quantitative Estimation of Alkaloids. C. Kippenberger. (*Zeit. anal. Chem.*, 1896, xxxv., 407-421.)—This is based on the fact that the compounds formed on treating alkaloids with iodised potassium iodide are soluble in acetone, and that on adding an aqueous solution of an alkaline hydroxide, alkali-metal iodide and iodate are formed, while the alkaloid remains in solution as hydriodide. The addition of an acid liberates the iodine from the iodate, and on titrating this free iodine with thiosulphate, an acidified aqueous solution of the alkaloid salt is obtained, from which, after evaporation of the acetone on the water bath and addition of an alkali, the alkaloid can be extracted with ether or chloroform (preferably the latter). Experiments with weighed quantities of the most commonly occurring alkaloids gave absolutely quantitative results when estimated by this process.

In the isolation of alkaloids from cadaveric remains, etc., the reactions for the alkaloids are often masked by the presence of other bodies, such as amines, amido acids, proteids, etc. In such cases the author extracts the supposed alkaloid by one of the usual processes, neutralizes the acidified solution, and adds a solution of iodine in potassium iodide, containing 12·7 grammes of iodine and 60 grammes of potassium iodide per litre, and as much more of the latter as is required to completely dissolve the iodine. After several hours, the precipitate is collected on an asbestos filter, washed several times with cold water, and dissolved in acetone. The solution is first made alkaline, then acidified, and then mixed with water, and the nearly colorless

liquid then contains the alkaloid as an acid salt. The acetone is evaporated in the water-bath, a little sodium thiosulphate solution added to the still warm liquid, then a slight excess of sodium carbonate, and the alkaloid extracted with chloroform or ether. With morphine or narceine a different method of extraction must be used, viz., amyl alcohol or chloroform from an ammoniacal solution, or chloroform with alcohol from an alkaline carbonate solution. Since glucosides do not react with iodine in potassium iodide, the method offers a means of separating them from alkaloids. The author's experiments on mixtures of alkaloids with the various substances likely to occur in decomposing organic matter show the reliability of the method.

The method is similarly applicable to the estimation of alkaloids in plant extracts. The extract is dissolved in acidified water, neutralized, and the alkaloid precipitated with iodine in potassium iodide. The precipitate is collected, preferably on asbestos in a Gooch's crucible, well washed with cold water, and dissolved in as little acetone as possible. The solution is treated with acid and alkali as before, then mixed with water, and shaken twice with petroleum spirit (B.P., 30° to 50° C.), which removes most of the acetone as well as impurities possibly present. The alkaloid is then extracted from the aqueous solution after the addition of sodium thiosulphate and sodium carbonate, as described above.

In order to prevent the alkaloid becoming brown during the evaporation of the solvent on the water-bath, the author recommends the presence of a trace of alkali. This is introduced by adding ether, which has been shaken with sodium carbonate, to the extraction solvent, and the error thus caused may be neglected, since that introduced by the variation in the amounts of water of crystallization of alkaloids is often greater. Ammonia is not suitable for the purpose, since it is volatile during the drying.

Objection is taken to the common method of isolating pure alkaloids by adding acid to the ethereal solution of the alkaloid bases, on the ground that when ammonia has been used to set free the alkaloid, the ammoniacal salt has been simultaneously precipitated. Moreover, in the case of old cadaveric matter, the salts of amido bases will also be separated. C. A. M.

Estimation of Alkaloids by Titration with Iodine Solution III. C. Kippenberger. (*Zeit. Anal. Chem.*, 1896, xxxv., 422-471.)—The author has continued his investigation on this subject (*cf.* ANALYST, xx. 201, and xxi. 191), and has arrived at the following conclusions:

1. For the accurate volumetric estimation of alkaloids in general in aqueous solution by means of iodine solution, it is necessary to standardize the latter against a weighed quantity of the given alkaloid. The most suitable strength for the iodine solution is $\frac{1}{10}$ or $\frac{1}{20}$ N. (This is a recapitulation of the conclusion drawn in the author's second paper—ANALYST, xxi. 192.)

2. The salts of strychnine and brucine, however, in aqueous solution react according to the equation $Alk.HCl + KI + I_2 = Alk.HI.I_2 + KCl$, when there is an excess of iodine, and 1 molecule of the alkaloid corresponds to 2 molecules of iodine. The best strength is $\frac{1}{20}$ N. iodine solution in 6 to 8 per cent. potassium iodide.

3. Morphine also may be titrated as in (2), provided the amount of potassium

iodide be increased. It is preferable to replace potassium iodide by potassium bromide, which is added in the solid form to the neutral morphine solution until a white precipitate appears. The iodine solution is then added, and, after standing for some time, the excess of iodine titrated with standard thiosulphate. Morphine may also be estimated in as neutral as possible solution of the sulphate by adding an excess of barium iodide and then the iodine solution. Accurate results may be obtained with quinine in aqueous solution by adding a large amount of potassium bromide, and using the iodine solution in only slight excess.

4. All the alkaloids may be accurately titrated by dissolving them in a known quantity of hydrochloric or sulphuric acid (the solution being as concentrated as possible), adding a calculated amount of silver iodide, then the iodine solution, filtering after two to three minutes, and titrating the excess of iodine in an aliquot portion of the filtrate. The silver iodide solution is prepared by dissolving 1·0 gramme $AgNO_3$ and 10 grammes of KI in 20 c.c. of water. The iodine solution $\frac{1}{20}$ N. To calculate the amount of silver iodide solution required, the factors 4·7 and 3·5 are employed for hydrochloric and sulphuric acids respectively, and the result when using this strength of silver iodide multiplied by 20. For example: 0·1 gramme of narcotine dissolved in 10 c.c. of water, containing 0·5585 per cent. hydrochloric acid, 0·05585 gramme $HCl \times 4{\cdot}7 = 0{\cdot}262495$ gramme AgI and this $\times 20 = 5{\cdot}2$ c.c., the amount of silver iodide solution required to convert the alkaloid hydriodide into the periodide. An excess of silver iodide must be avoided, but should it have occurred, the double salt of the alkaloid and silver which may precipitate may be dissolved by adding more potassium iodide. The details of the manipulation are the same as those given by the author in his first paper (see ANALYST, xx., 203, *Iodine Solution*). In the case of quinine, it must not be lost sight of that the compound $C_{20}H_{24}N_2O_2(HI)_2.I_4$ is formed, and that therefore 4 equivalents of iodine correspond to 1 molecule of the alkaloid. C. A. M.

The Estimation of Acetone in Urine. **G. Argenson.** (*Bull. Soc. Chim.*, 1896, 1055-1058.)—In this method, which is based on Léoben's reaction, the acetone is separated by distillation from the urine, transformed into iodoform by means of iodine and potash, the iodoform treated with alcoholic potash, and the potassium iodide formed titrated with decinormal silver nitrate solution.

On distilling urine containing acetone, the whole of the latter passes over in the first quarter of the distillate. To obviate the frothing somewhat, a little vaseline may be added, but constant attention is required.

In making a determination, 200 c.c. of the urine are distilled, and the first 50 c.c. of the distillate collected. To this are added 10 c.c. of an aqueous solution of potash (23° Bé., S.G. 1·186), and 5 c.c. of an iodine solution prepared by dissolving 105 grammes of iodine in a litre of water containing 180 grammes of potassium iodide.

The precipitate is collected on a filter, washed until the washings give no opalescence with silver nitrate, and introduced into a flask containing 20 c.c. of a concentrated alcoholic solution of potash free from haloid salts. The filter-paper is freed from the small amount of iodoform clinging to it by shaking with a mixture of ether

and alcohol in a stoppered flask, the solution being subsequently added to the main bulk.

After being boiled for several minutes to complete the reaction, the liquid is cooled, neutralized with acetic acid, and diluted to 200 c.c. An aliquot portion is then titrated with decinormal silver nitrate, potassium chromate being used as indicator, and from the number of c.c. used the weight of acetone is calculated.

The reactions involved are:

(1) $CH_3.CO.CH_3 + 3I_2 + 4KOH = CHI_3 + CH_3COOK + 3KI + 3H_2O$

and

(2) $CHI_3 + 4KOH = 3KI + HCOOK + 2H_2O$.

According to the first equation, one molecule of acetone should give one molecule of iodoform; but in practice the quantity obtained is always less than that required by theory, and is not proportional to the amount of acetone in the solution. It is, therefore, essential to always work under identical conditions; and for use with his exact mode of proceeding the author has constructed the following table of reference, in which is given the weight of acetone corresponding to the volume of silver nitrate solution used.

$\frac{N}{10}$ Silver Nitrate, c.c.	Grammes of Acetone per litre of Urine.	$\frac{N}{10}$ Silver Nitrate, c.c.	Grammes of Acetone per litre of Urine.
0·5	0·033	20	1·118
1	0·071	21	1·170
2	0·133	22	1·221
3	0·200	23	1·272
4	0·262	24	1·323
5	0·317	25	1·374
6	0·372	26	1·425
7	0·424	27	1·476
8	0·476	28	1·527
9	0·523	29	1·578
10	0·570	30	1·629
11	0·626	31	1·680
12	0·682	32	1·731
13	0·738	33	1·782
14	0·800	34	1·832
15	0·854	35	1·882
16	0·908	36	1·933
17	0·962	37	1·983
18	1·014	38	2·033
19	1·066		

C. A. M.

New Colour Reaction of Colza Oil. Palas. (*La Nature*, August 22, 1896; through *Ann. de Chimie Analyt.*, vol. i. [22], p. 434.) — The rose-red coloration produced by agitating colza oil with rosaniline bisulphite is peculiar to this oil; olive oil forms a more or less white emulsion, whilst sesame, cotton-seed, arachis, ricinus, and almond oils are decolorized, turnip, nut, and poppy oil remain unchanged, and linseed oil forms a bright yellow emulsion. The fatty acids of colza oil give no coloration.

The reaction is sufficiently delicate to detect 2 per cent. of colza oil in olive oil.

To prepare the reagent, which should be colorless, 30 c.c. of a 1 per cent. solution of fuchsin, 20 c.c. of 34° B. (S.G. 1·308) sodium bisulphite, 200 c.c. of water, and 5 c.c. of sulphuric acid, are mixed together in the cold. C. S.

On the Rancidity of Fats. E. Spaeth. (*Zeit. anal. Chem.*, 1896, xxxv., 471-493.)—After a long experimental investigation on this subject, the full details of which are given in the original paper, the author has arrived at the following conclusions:

1. The rancidity of fats (lard) is an oxidation process, caused essentially by the action of light and atmospheric oxygen. The unsaturated acids (oleic) are first attacked with the formation of acids containing less carbon, and subsequently aldehydic substances and oxy-acids are produced.

2. During the course of oxidation and formation of free acids, the amount of volatile acids is considerably increased (*cf.* ANALYST, xxi., 298).

3. All the acids present have a share in the formation of the free fatty acids.

4. As the oxidation proceeds, the absorptive capacity (for iodine) of a fat correspondingly decreases, owing to the above-mentioned oxidation and decomposition of the unsaturated fatty acids and their polymerization. Fats thus oxidized have a considerably higher refractive index than have the normal fats, which must be attributed to the polymerization of the unsaturated fatty acids.

5. In general, fats which have become rancid have a higher melting-point than that shown by the fresh fats. C. A. M.

On the Iodine Number of Cocoa Butter. F. Filsinger. (*Zeit. anal. Chem.*, 1896, xxxv., 517-521.)—Referring to the figures published by Strohl (ANALYST, xxi., 231), the author suggests that the higher limit (41·7 per cent.) might have been due to the presence of a certain amount of free fatty acids in the samples, which would obviously absorb more iodine than the neutral fat. From the results of the examination of cocoa butter from all sources made in conjunction with R. Henking in 1889, he came to the conclusion that the iodine number varied from 33·5 to 37·5 per cent. C. A. M.

INORGANIC ANALYSIS.

Determination of Oxygen by Alkaline Pyrogallol. F. Clowes. (*Chemical News*, vol. lxxiv., p. 199.)—An evolution of carbon monoxide occasionally takes place when oxygen is absorbed by alkaline pyrogallol solution, which, unless determined by absorption with cuprous chloride, falsifies the estimation. This can be prevented by using a sufficient excess of potassium hydroxide. The following proportions are recommended: 160 grammes of potassium hydroxide and 10 grammes of pyrogallol in 200 c.c. of solution, made by dissolving the hydroxide in 130 c.c. of water, and adding the pyrogallol to this. C. S.

On the Standardization of Potassium Permanganate. E. Riegler. (*Zeit. anal. Chem.*, 1896, xxxv., 522.)—The alteration in the strength of a solution of oxalic acid is prevented by the addition of a sufficient quantity of sulphuric acid. A solution prepared by dissolving 9·9654 grammes of pure oxalic acid in about 500 c.c. of water, adding 50 c.c. of concentrated sulphuric acid and making up to a litre, did not change in the slightest degree after standing for a year. Twenty c.c. of such a solution correspond to 100 milligrammes of potassium permanganate. C. A. M.

On the Estimation of Sulphur in Inorganic Sulphides. P. Jannasch and O. Heidenreich. (*Zeit. f. anorg. Chem.*, xii., 5, 358.)—The paper describes the analysis of commercial stannic sulphide by the method previously suggested (ANALYST, xvii., 197). The sulphide is heated in a weighed tube, through which oxygen is passed, until the sulphur is driven over into the receiver, the tube with its contained stannic oxide being afterwards weighed when cool. The receiver may contain either 3 to 4 per cent. hydrogen peroxide, or a mixture of very dilute hydrochloric acid with excess of bromine. C. H. C.

Gravimetric Estimation of Selenium. A. W. Pierce. (*Zeit. Anorg. Chem.*, xii., 6, 409.)—To avoid the tedious and frequently incomplete precipitation of selenium by sulphurous acid, the author substitutes potassium iodide in an acid solution as the reducing agent. The following procedure is given as the best: The solution containing the selenium is diluted to 400 c.c., acidified with hydrochloric acid, and three grammes of potassium iodide added; the solution is then boiled for ten to twenty minutes, when the selenium will be precipitated as the black modification, and will be found to be entirely free from mechanically held iodine. The selenium thus thrown down is collected on an asbestos filter, washed and dried at 100° C. until constant in weight. The precipitation and filtration can be carried out in thirty minutes. Both selenious and selenic acids can be estimated as shown above, but in the case of selenic acid the iodine is not liberated until the solution is boiled; and in order to complete the reaction the solution must be boiled for some time. Although selenic acid cannot be determined in the presence of selenious acid, the method has the advantage that the total selenium in a solution can be rapidly determined, whether it is present as selenic or selenious acid. H. C. L. B.

Brucine as a Reagent for the Detection of Nitrous Nitrogen in Presence of Sulphites. P. Pichard. (*Comptes rendus*, vol. cxxiii., p. 590.)—Brucine gives, with hydrochloric acid, a vermilion to yellow coloration in presence of nitrites, and by this test $\frac{1}{2\,4\,0\,0\,0\,0}$ part of nitrous nitrogen can be detected in solution. It is suitable for use in the examination of such bodies—drainage-waters, etc.—as contain nitrites and sulphites, the latter of which interfere with the accuracy of the Picini, Tromsdorff, and Griess nitrogen reactions. The substance is tested for sulphides by lead acetate, and for sulphites by a strong acid, and, if sulphur dioxide is detected, the brucine test is applied in the following manner. A drop of hydrochloric acid is first

added, and then small portions of brucine. In five minutes or so a tint varying from vermilion red to bright yellow appears if nitrites are present.

Brucine gives no coloration with chlorine or hypochlorites, but the following salts of brucine—nitrate, chloride, acetate, and (especially) sulphate—will give a vermilion red with these bodies. C. S.

Arsenical Sulphuric Acid a Source of Error in the Naumann-Wagner Process for the Estimation of "Available" Phosphoric Acid. G. Loges and K. Mühle. (*Chem. Zeit.*, 1896, xx., 984.)—Naumann's process for estimating the phosphoric acid in the Wagner extract of a slag—which consists in a destruction of the citric acid by means of 25 c.c. of sulphuric acid and 30 c.c. of strong nitric acid, followed by a direct precipitation with magnesia mixture—is so much quicker and less troublesome than the ordinary method that it is becoming very generally used. Occasionally some irregularities occur in the results obtained, and these appear to be due to the presence of arsenic in the sulphuric acid employed. In one instance, where the authors used an acid freed from nitrogen (for Kjeldahl estimations) by arsenious acid, they found 18·01 per cent. of available acid in a sample that only contained 17·68 per cent. of total phosphoric acid, the magnesium pyrophosphate being, of course, contaminated with the corresponding arsenate. F. H. L.

Detection of Fluorine in Silicates and Borates. J. A. Reich. (*Chem. Zeit.*, 1896, xx., 985.)—The material is moistened with sulphuric acid and placed in a crucible, which is covered with a watch-glass, convex side down, having a drop of water on its under surface. If any fluoride of silicon or boron is given off, a white layer of silica or boric acid will be found at the edge of the drop, and, after evaporation, will appear as an etched line. The two substances may be distinguished by treatment of the deposit with water, and by the turmeric-paper test for boric acid. F. H. L.

Estimation of Boric Acid. Schneider. (*Chem. Zeit.*, 1896, xx., 822.)—This process depends on the fact that boric acid is completely volatilized on distillation from an alcoholic solution. The material to be analysed is extracted under a reflux condenser with boiling absolute alcohol, the liquid cooled and filtered, and the residue washed with the same solvent. Raw flesh, however, is preferably cut up into fragments, and cooked in a well-covered beaker immersed in a water-bath. The mass is then cooled, rubbed down to a powder, an equal weight of anhydrous sodium sulphate added to absorb the water, and the whole extracted with alcohol as before. The solution is distilled from a flask immersed up to its neck in the water-bath till all the alcohol has passed over; and if turmeric paper shows that the last drops of the distillate still contain boric acid, more alcohol is introduced into the flask and the distillation repeated. A quantity of freshly-melted sodium carbonate is dissolved in water, and the alcoholic solution added gradually. The mixture is evaporated to dryness, the residue weighed, fused, and weighed again, the boric acid being calculated from the loss.

F. H. L.

The Solubility of Barium Sulphate. F. W. Küster. (*Zeit. f. anorg. Chem.*, xii., 4, 261-271.)—The author criticises the methods employed, and combats the conclusions arrived at, by Fresenius and Hintz, as set forth in their paper on "The Abnormal Solubility Ratio of Barium Sulphate" (*Zeit. f. anal. Chem.*, xxxv., 170-183). The results they obtained are not only at variance with the author's own experiments, but also with the observations of Kohlrausch and Rose (*Zeit. f. phys. Chem.*, xii., 241), and of Hollemann (*ibid.*, xii., 130), who employed more exact methods. The author concludes that the figures given by Fresenius and Hintz for the solubility of barium sulphate in pure water are incorrect, their solutions being obviously supersaturated, and gives 1 in 425,000 for the solubility in pure water at 18·3° C. (Fresenius 1 in 100,000, Kohlrausch and Rose 1 in 436,700, and Hollemann 1 in 429,700). He also shows that the solubility figures for barium sulphate in solutions of salts and in acids are in no way abnormal, but are quite in accordance with the modern theory of solution. C. H. C.

The Examination of Commercial Thorium Nitrate and Separation of Thorium from Cerium. R. Fresenius and E. Hintz. (*Zeit. anal. Chem.*, 1896, xxxv., 525-544.)—The authors examined 11 incandescent mantles, manufactured between March and October, 1895, which consisted principally of thoria (96·42 to 99·06 per cent.) and ceria (0·38 to 2·02 per cent.) with the object of determining whether the ceria was an accidental impurity in the thorium nitrate used, or had been intentionally added.

The sources of thorium nitrate are monazite, containing about 4 to 5 per cent. of thoria and 50 to 60 per cent. of oxides of the cerium group (ceria, neodidymia, lanthana); and Norwegian thorite or orangite, in which the thoria varies very greatly (often being 50 per cent.), but in which the percentage of the other earths, yttria, ceria, etc., is invariably small. In the preparation of thorium nitrate the purification of thorite is therefore considerably easier than that of monazite.

Three specimens of thorium nitrate, in use during March to October, 1895, one of which had been prepared from monazite, were analysed in the following manner: Twenty grammes of the thorium nitrate were dissolved in about 3 litres of water, sodium thiosulphate added, and the liquid boiled. The precipitate was filtered off, washed, and dissolved in hydrochloric acid. Any sulphur remaining undissolved was burned off, the ash fused with acid potassium sulphate, dissolved in dilute hydrochloric acid, and precipitated with ammonia. The precipitate was washed, dissolved in hydrochloric acid, and the solution added to the main solution. The whole was evaporated to dryness, the residue taken up with water and a few drops of hydrochloric acid, and again precipitated with sodium thiosulphate. The precipitate was filtered off and washed.

The filtrates from the first and second thiosulphate precipitations were treated with ammonia, the precipitates dissolved in hydrochloric acid, the solutions united, evaporated to dryness, and the residue taken up in slightly acidified water and again boiled with sodium thiosulphate. This treatment was again repeated. The filtrates from the two last thiosulphate precipitates were precipitated with

ammonia, the precipitates washed, dissolved in nitric acid, and the united solutions again precipitated with thiosulphate. The filtrate was treated with ammonia, and the resulting precipitate filtered off, washed, and dissolved in nitric acid. The solution was evaporated to dryness, the residue taken up with water, and precipitated, warm, with oxalic acid.

The precipitate (containing cerium, yttrium, lanthanum, and neodidymium) was ignited and weighed, fused with acid potassium sulphate, dissolved, ammonia added, and the precipitate filtered off, washed, and dissolved in hydrochloric acid. The solution was nearly neutralized with sodium carbonate, and boiled after the addition of sodium acetate, acetic acid, and sodium hypochlorite. The precipitate was filtered off, washed, dissolved in hydrochloric acid, and again precipitated in the same way. It was then washed, dissolved in nitric acid, precipitated with ammonia, filtered off, washed, ignited, and weighed as cerium oxide.

The filtrate was acidified with hydrochloric acid, boiled, and decomposed with ammonia. The precipitate was filtered off, washed, and dissolved in hydrochloric acid. The solution was evaporated to dryness, the residue taken up in a few drops of water, and precipitated by means of a saturated solution of potassium sulphate. After standing some time the precipitate was filtered off, and washed with the saturated solution of potassium sulphate, dissolved in water containing a few drops of hydrochloric acid, precipitated with ammonia, filtered off, washed, and dissolved in nitric acid. The solution was evaporated to dryness, the residue taken up in water, precipitated with oxalic acid, and the precipitate collected, washed, ignited, and weighed. It contained the neodidymium and lanthanum oxides.

The filtrate from the potassium sulphate precipitate was precipitated with ammonia, washed, dissolved in nitric acid, and again precipitated with ammonia, washed, ignited, and weighed as yttrium oxide. Thorium oxide was estimated by difference.

The results thus obtained were:

	Thorium Nitrate (from Monazite). E. Merck. August, 1895. Per cent.	Thorium Nitrate. E. de Haën. July, 1895. Per cent.	Thorium Nitrate. E. de Haën. March, 1895. Per cent.
Thoria	47·2600	46·2066	44·9410
Ceria	0·0885	0·0463	0·0910
Neodidymia (including lanthana)	0·0940	0·0521	0·0665
Yttrium oxide ...	0·1430	0·0373	0·0070
Nitric acid, water, and acids not estimated	52·4145	53·5823	54·8945
Zironia	—	trace	—
Lime, magnesia, iron silica	—	0·0754	—
	100·0000	100·0000	100·0000

Calculated on the substance left after burning in an incandescent mantle, these corresponded to:

	Per cent.		Per cent.		Per cent.
Thoria	99·817	...	99·546	...	99·635
Ceria	0·186	...	0·100	...	0·202
Neodidymia (including lanthana)	0·197	...	0·112	...	0·147
Yttria	0·300	...	0·080	...	0·016
Zirconia	—	...	trace	...	—
Lime, magnesia, iron silica	—	...	·162	...	—
	100·00		100·000		100·000

Since the amount of ceria found in the 11 mantles (average 1 per cent.) was about five times as great as that contained in the most impure of the samples of thorium nitrate when calculated on the total quantity of oxides that would be left in a mantle after burning, the authors concluded that either the ceria was intentionally added, or that the thorium nitrate used was of a much less degree of purity than was attainable in 1895.

To confirm this view, they made mixtures containing 2·02 and 0·38 per cent. of ceria, the highest and lowest limits found in the eleven mantles, and showed that by precipitating the solutions three times with sodium thiosulphate, and then precipitating the filtrates with oxalic acid as described above, from 97 to 98 per cent. of the cerium oxide taken could be recovered, so that the methods known to chemists in 1895 were quite sufficient to effect a far greater amount of separation of cerium from thorium than was the case in the mantles analysed. C. A. M.

The Separation of Thorium from the other rare Earths by means of Potassium Trinitride. L. M. Dennis. (*Jour. Amer. Chem. Soc.*, 1896, xviii., 947-952.)—It is shown that thorium can be quantitatively separated from the other rare earths by adding a solution of potassium trinitride to a neutral solution of the rare earth salts. This reaction may be attributed to the weak basicity of thorium, and the author's experiments show that it may be represented by the equation

$$Th(NO_3)_4 + 4KN_3 + 4H_2O = Th(OH)_4 + 4KNO_3 + 4HN_3.$$

The reagent is prepared by neutralizing a dilute solution of hydrazoic acid with a dilute solution of pure caustic potash, and then adding sufficient hydrazoic acid to give a distinctly acid reaction.

In the test experiments with thorium alone, a solution of pure thorium chloride was employed which contained in 1 c.c. an amount corresponding to 0·00521 gramme of ThO_2, as determined by precipitation with ammonia. On adding to this solution a few c.c. of potassium trinitride solution, and boiling for one minute, a flocculent precipitate was obtained. This was washed by decantation with hot water, transferred to a filter, ignited, and weighed as ThO_2. The amount of thorium dioxide obtained was 0·00520 gramme in 1 c.c. Some of the results with mixtures of pure salts of thorium, cerium, lanthanum, and didymium are given in the following table.

		Taken.	Found.
I.	Thorium	0·1300	0·1294
	Ce., La., Di., oxides	0·0332	—
III.	Thorium	0·0785	0·0783
	Ce., La., Di. oxides	0·2490	—
VII.	Thorium	0·0570	0·0588
	Ce , La., Di. oxides	0·8300	—

C. A. M.

A Sensitive Form of the Diiodide Reaction for the Detection of Mercury. **P. Jannasch.** (*Zeit. f. anorg. Chem.*, xii., 2, 143-145.)—For the ready recognition of minute traces of mercury the author operates as follows: Deposit the mercury on copper foil by warming in a slightly acid solution, pour off the solution, wash the metal with cold water, and dry with a minimum of friction by means of blotting-paper. Put the amalgamated copper at the bottom of a long narrow test-tube, remove the last traces of water by sucking air out of the tube. Draw out the latter in the middle to capillary bore. By careful heating drive the mercury into the shoulder under the constriction, divide the tube at the drawn-out part, cut off the closed end, and place the portion containing the sublimate in a cylinder containing a small fragment of iodine. The mercury, when converted into iodide, appears scarlet in thick and yellow in very thin layers. Half a milligram of mercuric chloride yielded a sublimate of mercury which was practically invisible, but was easily distinguished when converted into iodide.

A still more delicate method is to place the mercury-covered slip of copper foil on a watch-glass close to, but not touching, a small fragment of iodine. Cover with another watch-glass, and allow to stand for ten minutes. A deposit of mercuric iodide of the same shape as the foil is formed on the glass, and may be further identified by the microscope. In this way 0·01 milligram of mercuric chloride may be detected.

C. H. C.

The Separation of Mercury from Tin. **P. Jannasch and H. Lehnert.** (*Zeit. f anorg. Chem.*, xii., 2, 132, 133.)—The same apparatus is used as for the estimation of sulphur in sulphides (ANALYST, xvii., p. 197).

Fill the receiver with dilute nitric acid, dissolve the mixed salts in strong nitric acid, evaporate off the excess of acid, dilute to about 300 c.c., add 5 c.c. concentrated sulphuric acid, heat gently, and precipitate with hydrogen sulphide. Wash the precipitate with warm water, dry at 90° C. Detach the precipitate from the filter as thoroughly as possible, cut up the latter into small pieces, and burn in the combustion tube with suitable precautions for avoiding the distillation of tarry products, cool the apparatus, add the main bulk of the precipitate and burn this also. Mercury distils over, while stannic oxide remains behind, and can be weighed. Dissolve the mercury in the receiver by evaporating the combined liquids and adding sulphuric acid. Remove nitric acid as far as possible by evaporation, dilute largely with water, and precipitate the mercury as sulphide; filter, wash, dry and weigh. C. H. C.

The Separation of Mercury from Antimony, Copper, and Arsenic. P. Jannasch. (*Zeit. f. anorg. Chem.*, xii., 5, 359-364.)

(1) *Mercury from Antimony.*—The metals are precipitated as sulphides, filtered off, washed, and dried at 95° C. The precipitate is removed from the filter and placed on one side, the filter-paper cut into strips, rolled up, and placed in the tube of the apparatus previously described (ANALYST, xvii., 197), the receiver of which is filled with dilute nitric acid. Fuming nitric acid is then gradually added until the tube is about a quarter full, when it is immersed in hot water (or heated very carefully over a naked flame) until solution is complete. The excess of acid is evaporated off by heating to 90° or 100° C. (in a phosphoric acid bath) in a current of dry air or carbonic acid. The black residue is treated with the same volume of acid and dried once more, then the precipitate introduced, washing in the last particles with dilute nitric acid. Concentrated (ordinary) nitric acid is added, dried as before, and evaporated twice with the fuming acid, finally raising the temperature to 150° to 180° C. The tube is then connected with the oxygen supply and the receiver, and heat applied, beginning at the receiver end, till all the mercury is expelled from the tube. When the apparatus is quite cold, the residual SbO_2 may be weighed direct, or may be converted into sulphide by fusion with sulphur iodide in a stream of sulphuretted hydrogen, subsequently driving off the excess of sulphur, and heating the residual Sb_2S_3 in a stream of bromine (see *Berichte*, xxvi., 1422, *Zeit f. an. Chem.*, ix., 201). The mercury is estimated in the receiver in the manner already described.

(2) *Mercury from Copper.*—The metals are precipitated as sulphides, washed, and dried, the precipitate separated from the filter, and the latter burnt in the weighed combustion-tube in a stream of oxygen The precipitate is then introduced and heat applied, at first gently, and then more strongly, a stream of oxygen being maintained in the tube until all the mercury is driven over into the receiver (which contains a mixture of dilute nitric acid and bromine water). Weigh the residual cupric oxide directly by heating the tube till constant in weight, or dissolve in hydrochloric or nitric acid and precipitate with soda, etc. Estimate the mercury as before.

(3) *Mercury from Arsenic* —The nitric acid solution of the mixture is introduced into the combustion-tube of the apparatus and dried by heating in a phosphoric acid bath in a current of dry air. The residue is then taken up with a drop of water, 0·6 gramme of magnesia free from chlorine added, well mixed, and again evaporated to dryness, the temperature being finally raised to 180° C. After removing the tube from the phosphoric acid, it is cleaned externally, connected with the oxygen supply, and heated until all the mercury is expelled into the receiver (containing dilute nitric acid and 15 c.c. hydrogen peroxide). The tube is allowed to cool gradually, the residue dissolved in 5 c.c. concentrated nitric acid, transferred to a beaker, 0·3 gramme of citric acid added and excess of ammonia, and the arsenic estimated as magnesium arseniate or ammonium magnesium arseniate. C. H. C.

Notes on Reinsch's Test for Arsenic and Antimony. J. L. Howe and P. S. Mertins. (*Jour. Amer. Chem. Soc.*, 1896, xviii., 953-955.)—The fact that arsenious oxide and antimonious oxide (Sb_2O_3) crystallize in isomorphous forms, has led to the

conjecture that the antimonious oxide subliming from the copper might sometimes be mistaken for arsenic. To settle this point, the authors have made a large number of determinations with different salts of the two substances, and with the various organs of two cats poisoned by tartar emetic. The degree of heat required to sublime the antimony was perceptibly higher than in the case of arsenic, and out of 185 tests not one yielded a sublimate of antimonious oxide showing under the microscope any trace of crystallization. They conclude, therefore, that this corroborative negative evidence must be considered to have the weight of positive evidence.

With reference to the substances tested the following notes are recorded: (1) All arsenious compounds soluble in hydrochloric acid give the deposit on copper immediately on heating. (2) Commercial "metallic" arsenic readily gives a deposit. (3) Freshly-sublimed "metallic" arsenic (bright crystals) gives no deposit. (4) Arsenates deposit only after several minutes' boiling. (5) In the presence of nitric acid or chlorates no deposit is obtained, owing to the copper dissolving. (6) When aqua regia or potassium chlorate have been used to dissolve the arsenic compound, the solution should be evaporated to dryness with hydrochloric acid, and tested, as in the case of arsenates. (7) Presence of organic matter does not affect the test. (8) If much arsenic be present, only a small portion of the substance should be taken, as when a large amount of arsenic is deposited on the copper it does not adhere with firmness. (9) Antimony is not deposited as rapidly as arsenic, and the deposit has a violet tint, very distinct from the iron-gray deposit of arsenic. C. A. M.

On the Separation of Bismuth from the Metals of the Copper and Iron Groups. P. Jannasch and S. Grosse. (*Zeit. f. anorg. Chem.*, xii., 5, 398.)—The authors, in the course of a large number of separations of arsenic from the metals of the copper and iron group by their dry distillation process (*Zeit. f. an. Chem.*, ix., 274), have observed that bismuth, like tin, may be readily volatilized in a stream of hydrochloric acid, and found on this reaction a process for the separation of tin from the other metals of the above-mentioned groups. C. H. C.

Analysis of "Cap Composition." F. W. Jones and F. A. Wilcox. (*Chemical News*, vol. lxxiv., p. 283.)—Having found that mercury fulminate is soluble in acetone saturated with ammonia, the authors have utilized this property for separating the fulminate from antimony sulphide and potassium chlorate in the analysis of "cap composition."

The carefully-ground substance is placed on a tared filter-paper (previously moistened with the solvent and inserted in a tapped funnel) and exposed to the action of acetone saturated with ammonia for three or four hours. It is then washed with the same solvent until the washings no longer give coloration with ammonium sulphide, and afterwards washed with acetone until the washings leave no residue or evaporation. The residue is dried and weighed; the loss in weight represents the mercury fulminate. The potassium chlorate is then extracted by washing with water, the antimony sulphide being left behind.

Two analyses of mixtures of known composition are given, viz.:

	A.		B.	
	Percentage taken.	Percentage found.	Percentage taken.	Percentage found.
Antimony sulphide ...	36·47	36·25	37·34	37·22
Potassium chlorate ...	33·25	33·71	46·03	46·43
Mercury fulminate ...	30·27	30·02	16·61	16·34

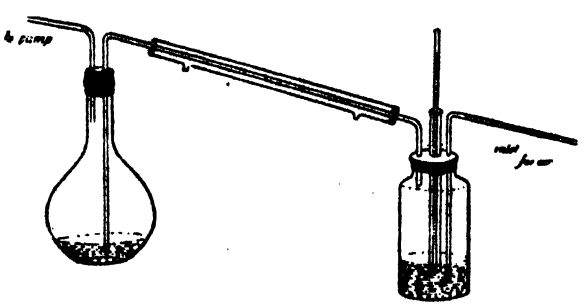
C. S.

A New Method of Converting Sulphates into Chlorides. P. Jannasch. (*Zeit. f. anorg. Chem.*, xii., 3, 223-224.)—The author points out that the method (ANALYST, xxi., 51), of decomposing silicates by means of boric acid is suitable for the conversion of sulphates into chlorides, as during the fusion all the sulphuric acid is expelled, and its place is taken by the hydrochloric acid subsequently employed for the solution of the melt. C. H. C.

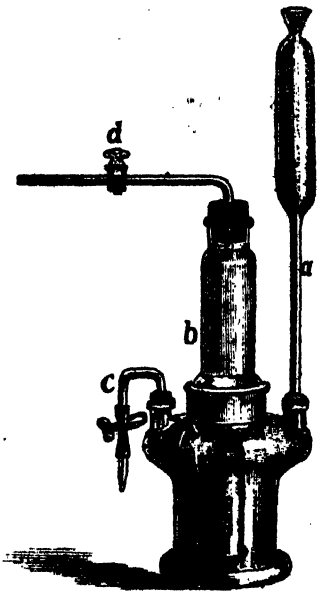
Iodimetric Method for the Determination of Carbon Dioxide. J. K. Phelps. (*Zeit. f. anorg. Chem.*, xii., 6, 431.)—This method consists of the absorption of carbon dioxide by a cold saturated solution of barium hydroxide; the excess of barium hydroxide is then neutralized by iodine, and the excess of iodine added is titrated with a standard solution of arsenious acid. H. C. L. B.

APPARATUS.

A Simple Method for Obtaining a Low Temperature. C. E. Sage. (*Pharm. Journ.*, 1896, p. 397.)—The apparatus consists of a 6-oz. wide-mouthed bottle fitted with a cork pierced with three holes, one of which is large enough for a test-tube, and the others for an inlet and outlet air-tube. The inlet tube passes nearly to the bottom of the flask, while the other passes only a short distance through the cork, and is connected at the other end with a condenser. The flask is partially filled with ether, and the sample to be examined (oil, etc.) is placed in the test-tube, together with a thermometer. Air is now forced or drawn through the ether, which, as it evaporates rapidly, cools the tube, and a temperature of from - 4° to - 5° C. can be easily maintained for a long period. Most of the evaporated ether is retained by the condenser, and the air leaving the condenser can be washed by bubbling through a flask of water before being allowed to escape. C. A. M.



An Improved Laboratory Gas Apparatus. R. Barge. (*Chem. Zeit.*, 1896, xx., 955.)—As shown in the accompanying illustration, this apparatus possesses several advantages over the ordinary Kipp's generator. The portion *b* is closed at its narrowest part by a perforated rubber stopper, on which the substance to be decomposed rests. Just below this point, and near the level of the acid in the lower vessel, a small hole is made in the side, so that while the spent liquid passes down to the bottom, the fresh acid is forced by the pressure given by the tube *a* through this aperture, and therefore reaches the solid without contamination. It is claimed that 94 per cent. of the acid required to work the apparatus is thus made available. The base of the tube *a* is cut off slightly below the level of the stopper; consequently a small quantity of liquid only rises into *a* when the cock *d* is shut, and the stability of the apparatus is not impaired.



The author also remarks that the ordinary Kipp can be improved in this latter respect by cutting off the long central tube just below the top of the bottom bulb, and by replacing the usual stopper by a tube and clip similar to the one shown at *c*. F. H. L.

MISCELLANEOUS.

Alcoholic Fermentation. Eduard Buchner. (*Ber. d. d. Chem. Gesell.*, 1897, p. 117.)—The author has succeeded in obtaining an extract from yeast which apparently possesses the power of causing alcoholic fermentation. One kilogramme of pressed brewers' yeast was triturated with an equal weight of quartz-sand and 250 grammes of kieselguhr until a plastic mass was obtained. With this 100 c.c. of water were intimately mixed, and the whole submitted to a pressure of 400 to 500 atmospheres in a hydraulic press. The resulting cake was then powdered, another 100 c.c. of water mixed with it, and the treatment repeated. In this way 500 c.c. of an extract were obtained, which, after being passed through a Berkefeldt filter, was able to induce fermentation in solutions of maltose, glucose, lævulose, or cane-sugar. The yeast extract was a slightly opalescent fluid of a pleasant yeasty odour, which had a specific gravity of 1·0416. On heating it a coagulum began to appear at 35° to 40° C., and on boiling the fluid assumed a semi-solid consistence. Buchner considers that the fermentative action is probably caused by an enzyme present in the yeast extract, but this he has not yet been able to isolate. W. J. S.

THE ANALYST.

MARCH, 1897.

PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS.

THE monthly meeting of the Society was held on Wednesday evening, February 3, in the Chemical Society's Rooms, Burlington House, the President, Dr. Bernard Dyer, occupying the chair.

The minutes of the previous meeting were read and confirmed.

The following gentlemen were proposed for election: as members—Mr. John C. Candy, B.A., B.Sc. (Lecturer on Chemistry at the London Hospital); Henry de Mosenthal, F.I.C. (Chemist to Nobel's Explosives Company); John H. B. Jenkins, (Chemist to the Great Eastern Railway Company); Fredk. W. Harris (Public Analyst for the Borough of Burnley, Lancs.). As associate—Edward Halliwell (assistant to Mr. T. Fairley, Leeds).

The following gentlemen were elected associates of the Society: Arthur French Angell (assistant to Mr. Arthur Angell); W. H. Jackson (assistant to Mr. A. C. Wilson); Arnold Rowsby Tankard (assistant to Mr. A. H. Allen).

The following papers were read:

"Some Analyses of Water from an Oyster Fishery." By Charles E. Cassal.

"The Composition of Meat Extracts and Similar Products." By Otto Hehner.

"The Distillation of Formaldehyde from Aqueous Solution." By Norman Leonard, B.Sc., Henry M. Smith, and H. Droop Richmond.

"Remarks on Formaldehyde." By Charles E. Cassal.

THE ANNUAL DINNER OF THE SOCIETY OF PUBLIC ANALYSTS.

This took place, after the annual meeting on January 13, at the Criterion, under the chairmanship of the newly-elected President, Dr. Bernard Dyer. About eighty members and guests were present, amongst the latter being Mr. T. H. Elliott (Secretary of the Board of Agriculture), Professor Thorpe, F.R.S., Mr. Hudson E. Kearley, M.P., Mr. A. G. Vernon Harcourt, F.R.S. (President of the Chemical Society), Dr. W. J. Russell, F.R.S. (President of the Institute of Chemistry), Mr. J. Fletcher Moulton, Q.C., F.R.S., Professor H. E. Armstrong, F.R.S., Professor J. Millar Thomson, Professor Dunstan, F.R.S., Professor Meldola, F.R.S., Mr. C. E. Groves, F.R.S., Mr. Walter Hills (President of the Pharmaceutical Society, Dr. Messel (Chairman of the London Section, Society of Chemical Industry), Mr. A. C. Tanqueray (President of the Institute of Brewing), Mr. Aubrey Rake, Mr. A. B.

Gripper (Secretary of the London Corn Trade Association), Mr. W. P. Ricketts, Mr. J. Innes Rogers, Mr. G. C. Barnes, and Mr. F. W. Beck.

The CHAIRMAN (Dr. Dyer), in proposing the toast of "The Queen," said that wherever that toast was drunk this year it would be received with special enthusiasm, on account of the fact that Her Majesty was now completing the sixtieth year of her reign. It was pleasant to remember that for more than one-third of that long period the members of this Society had been able to drink this toast together, and there were, he believed, one or two present besides himself who had been present on every one of these annual occasions. This year the toast would be drunk with even more enthusiasm than in the past, and he sincerely trusted that it might be many years yet before any change was required in the formula in which Englishmen expressed their loyalty (applause).

The CHAIRMAN, in proposing "The Houses of Parliament," said he would venture to linger somewhat longer over this toast, because, owing to the special circumstances of the past year, he regarded it as the toast of the evening. They had been looking forward to the honour of having with them the Earl of Winchilsea, but he was very sorry to have to tell them that he had received a telegram from Lord Winchilsea from Hastings, regretting at the last moment that his health did not allow him to travel up to town. Lord Winchilsea was a nobleman who was well known throughout this country for the sympathy which he had with distressed agriculture, and, he might add, for the untiring energy with which he expressed that sympathy in a practical form by doing all that lay in him to assist the cause of the suffering farmer. Had Lord Winchilsea been present that evening, he (the speaker) was sure they would have had from him, in reply to the first part of the toast, a speech to which they would have all listened with very great interest. They had, of course, no politics in that Society. No doubt many of them had individual views, and differing individual views, on such subjects as the fiscal policy of this country; but there was one form of protection about the justice of which every one of them was agreed, and that was protection for the British farmer against unfair competition in the way of adulterated food products. Upon them, as public analysts, largely devolved the duty of enforcing that protection, and it was to show his sympathy with them in their work that Lord Winchilsea was good enough to promise to come to them that evening. They were sorry that he was ill, and they were sorry to miss his speech. They had present a well-known member of the House of Commons, whose name would be received with enthusiasm, namely, Mr. Hudson E. Kearley. Mr. Kearley was a distinguished representative of English commerce, and his efforts towards the improvement of the Food and Drugs Acts were known to them all; and they had lately been allowed to do themselves honour by recognising these efforts in electing Mr. Kearley an honorary member of that Society. They also had present Mr. Fletcher Moulton, who had served his country well in the House of Commons, though, owing to the fortunes of political warfare and to the indiscriminating caprice of his constituents, he was at present taking a temporary—they hoped only a temporary—rest from his Parliamentary labours. It was hardly necessary to remind the company that a large and very representative committee of the House of Commons had sat for the last three sessions to consider in what direction their food legislation

might be modified or reformed. That committee had collected a vast mass of evidence, to which that Society had—he thought he might say without boasting in the presence of their guests—made very valuable contributions, partly through individual members, but more especially as a Society through the mouth of their valued and very honoured and hard-working Past-President, Mr. Otto Hehner; and he would here say that he was very pleased to be able to take this opportunity of publicly thanking Mr. Kearley for the very great assistance which he had given to them during that inquiry, in enabling them to put their evidence before the committee in a satisfactory manner. This committee had now made a very full and very valuable report. There might be minor details in that report with which they could not all personally agree; he would not go into this, for the retiring President, Dr. Stevenson, in the very able address which he gave them that afternoon, had carefully gone through that report and discussed it, he might almost say, clause by clause. But he was sure he might say, leaving aside the details, that their Society was, broadly and practically speaking, in cordial agreement with the recommendations of the committee, and, in the presence of a representative of the House of Commons, he ventured, on behalf of the Society, to express the hope that the recommendations of that committee, or many of them, might be speedily transformed into law. They were most anxiously looking out for a new Bill, and when that Bill found its way into Parliament, they would look to Mr. Kearley and to their other friends there to help them in carefully watching the Bill; to consider any suggestions which they as a Society might possibly have to make on it, and to see that no amendments might be introduced into that Bill which might even remotely threaten to lessen its efficiency, without thorough discussion and thorough consideration. He thought he was not saying too much when he said that they, as public analysts, had done a great deal of good work in the past; they hoped to do better work in the future, but they could not do this unless the legal and administrative machinery underwent some improvement. For this improvement they were now looking to Parliament, firstly, for the sake of the food consumer, the great public; secondly, for the sake of the honest trader; next, for the sake of the British farmer; and he might say, finally, for their own sakes as officers who wanted to do their duty in as thorough a manner as possible.

Mr. Hudson E. Kearley, M.P., said he was glad to have the privilege afforded him of responding to this toast, if only for the opportunity of thanking the Society of Public Analysts for the great honour they saw fit to confer upon him last year by electing him an honorary member of their distinguished body. He desired to say that he appreciated that distinction highly, and any work that he might have done that seemed, in their estimation, to have entitled him to this consideration at their hands, he was sure had been a work of pleasure, and the honour they had paid him in conferring this distinction upon him had been far more than any endeavour of his had deserved. He regretted that he was alone in replying to this toast. He would have liked to have heard the Earl of Winchilsea respond before himself for that august body of which he was a member, but he would have been especially interested in hearing him relate his experience, and the result of the contest that he had been carrying on with a great amount of success on behalf of distressed British agriculture. He would, no doubt, have referred to the necessity that he felt to exist for some amendment of the

laws, in order that agriculturists might be protected from what the Chairman had called unfair competition. He would, no doubt, in following Lord Winchilsea, have said what he was going to say now—that although this question of adulteration was a question that materially affected agriculture, it was not only an agricultural question, but a question that affected the commercial interests of this country outside the agricultural interest, largely—more largely, indeed, in his opinion, than it affected agriculture. Still, it was encouraging to find that there was a body of men like the analysts of this country looking forward, as those interested in commerce were, to some amendment of the law, in order that they might be protected from the unfair results that arose owing to the inefficiency of the present laws. The past year, in a Parliamentary sense, had been an important one to the Society of Public Analysts. It had been important by virtue of the fact that the committee for which the Society had been working so long, and the appointment of which was mainly owing to its efforts, had issued its report. He agreed with the Chairman that that report did not contain all that they might have wished, but still, he believed that it would mark the commencement of a new and brighter era, not only for the commercial interests of the country, but for the public analysts, who had felt, more directly, possibly, than had the other bodies interested in this subject, the handicapping conditions under which their professional work was carried out so long as the law remained in its present unsatisfactory condition. They were all looking forward to some harvest as the result of their efforts. Those efforts had been long and arduous, and at times some friction had arisen between the views as expressed by the public analysts and those who differed from them; but now the committee had reported they were naturally expecting to get some satisfaction in the shape of a Bill which they hoped would ultimately become an Act of Parliament. He would suggest that the Bill they hoped to see introduced should be one to supersede all the Acts of Parliament at present on the statute-book dealing with the subject of adulteration. Personally, he would hope to see an Adulteration Bill introduced that would cover the whole field, and not leave them, as they were now, with one measure to deal with one article only, another measure to deal with food and drugs—and only partially with those; but that they would have a comprehensive, intelligible measure, that analysts could understand, that traders could understand; and he might go further—that the judiciary of the land could understand also. He had said that this committee had led to some differences of opinion, some of which, no doubt, were very strongly expressed; but he thought everybody was agreed that the best policy to pursue now was to bury all differences and look forward to the happy days that appeared to be in front, subject to this Bill being introduced. The most important recommendation of the committee, so far as concerned the public analysts, was that there should be set up a court of reference. It would be almost impossible for any satisfactory outcome to arise from their endeavours unless certain vexed questions were to be settled from time to time by such a court of reference. He did not believe it was possible to put into an Act of Parliament everything that was needed by the commercial interests and the public analysts on some of these vexed questions. Taking, for instance, the question of standards: the very moment one suggested a standard one was met by the objection on the part of others that if a standard were set up it inevitably meant that every-

body in the country lowered their goods down to this standard. There was something to be said for that. But if they had this court of reference they would have, he hoped, a competent body that would be fully alive to the necessity of keeping pace with the adulterations that might spring up. It had been his pleasure and privilege on the committee to examine, on behalf of the public analysts, many of their witnesses. He might say that the witnesses they submitted to the committee were experienced men, and naturally highly intelligent. His friend Mr. Otto Hehner he would almost describe as a model witness. He was so full of information that a special Blue-book might well have been devoted to his evidence. Mr. Hehner favoured the committee with most excellent evidence, and he (the speaker) was sure that if they succeeded in putting into an Act of Parliament only one-half of his recommendations, they would have a very progressive Act indeed. He did not propose to go through the various recommendations of the committee, but as he was in the presence of two or more representatives of Government Departments, he was obliged to give expression to this thought, that they did really require a Government department to look after the interests of this question. At present they had the Board of Agriculture, a body that, as was pointed out before the committee, was interested mainly in the question as it affected agriculturists; but he did think that no particular interest should be singled out for preferential consideration, but that the whole of the interests involved—those of the agriculturist, those of the trader, and, above all, those of the consumer—should be considered together. It was difficult to suggest what that department should be, because each department, in its particular work, no doubt did yeoman service on behalf of its particular interest; but it appeared to him that it would be most advantageous if they could commit the responsibility of administering the new law, which he believed would be passed, to a department which made the question of adulteration a leading feature of its departmental work. It had been heard in evidence that the local authorities were very remiss in some districts in working the existing Acts of Parliament. It had been said that the amount of adulteration that was found, from samples that were analysed, bore a definite relation to the amount of activity that was exerted by the local bodies. They had been told that so few samples were taken in many districts that the present Acts were practically allowed to be a dead letter. He knew that the public analysts thought—and he himself certainly agreed—that it was necessary that some central authority should be responsible for seeing that these Acts were efficiently administered, but he regretted to say that the committee could not come to a favourable decision on that point. It was pointed out that it would be very difficult for a central authority to undertake the responsibility of seeing that the Acts were actively administered, and he supposed there was no going back on the recommendation of the committee in that direction, but, nevertheless, he did venture to hope, as he had already said, that to one Government Department would be entrusted the responsibility of working these Acts on behalf of all the authorities concerned. Any movement in this direction must, he thought, tend to be beneficial to all concerned. The various recommendations of the committee would be of service in drawing up a new Bill. He could not say, even unofficially, that they were to have a new Bill, but he could answer for this, that if the Government did not

recognise their work, and did not introduce a Bill, there were certain of them that were interested in this question who were prepared with a Bill which, he thought, would meet the views, at all events, of the great majority of those present. Personally, he was looking forward with some amount of interest to the declaration of the Queen's Speech. He thought that this subject was sufficiently important to entitle it to some recognition immediately Parliament opened, and, without in any way holding out any threat to the Government as to the action that would be taken, he thought that if the Queen's Speech did not show some recognition of this question by an expression of the intention of the Government to introduce a Bill, someone would be prepared to ask the reason why. Individually, he would desire to say this: that they recognised the position of the Government as rather more difficult than their own, for they themselves were in a position of no responsibility, and consequently had greater freedom. The Government, on the other hand, naturally had to be somewhat guarded in its action upon such a question, but he hoped that the Government would see its way to an early opportunity of introducing such a Bill as was wished for.

Mr. Hehner, in proposing the toast of "The Various Government Departments," said he believed there was no public officer who had relations with so many Government Departments as the public analyst. He was appointed by a County Council, and was nominally a County Council officer, but he was under the control of the Local Government Board; he had to look to the Inland Revenue Department for control; he had to report, in his character of district agricultural analyst (which meant as a public analyst under the Fertilizers and Feeding Stuffs Act), to the Board of Agriculture; and, if the recommendations of the Food Products Adulteration Committee were carried into effect, he would also in the near future have to do with the Board of Trade. In one way this extensive connection with Government Departments was a testimonial to the usefulness of the public analyst as an officer, but it was not without its dangers. One man had only a certain value, and if this one man was split between five authorities, the proportion of his total value allotted to each might be but small.

There were present the representatives of two important Government Departments with which public analysts were connected. It was not the first (and, he trusted, would by no means be the last) occasion upon which the members of the Society had had the honour and pleasure of seeing among them Mr. T. H. Elliott, the Secretary of the Board of Agriculture. Public analysts ought to be (but unfortunately were not always held to be) of importance, and ought to be of use to the agriculture of this country, not only in contending against illegitimate home and foreign competition, but in stimulating scientific agriculture in many of its aspects. The public analyst should have been in past years in a position to exercise a useful influence in raising the standard of production of agricultural produce of all kinds, and also in relation to many scientific questions in which the agriculturists of this country were concerned. Unhappily, however, this had not been so altogether in the past, but they could look forward in the future to receiving able assistance from the Board of Agriculture. A great many public analysts were also district analysts under the Fertilizers and Feeding Stuffs Act, but they had not been able to be of much use in this capacity; but they looked forward, however, to an amendment that would increase the efficiency of the

Act, which, although well meant, had been lying, practically speaking, dormant. He thought he was rightly informed that the time was not far distant when an amendment would be introduced which would render the Act not merely a paper Act, but a really practical, workable measure.

As to the Inland Revenue Department, with which public analysts were so closely connected, he welcomed, in the name of all his colleagues, both present and absent, Professor Thorpe. It was particularly agreeable to him, for reasons which he need not specify, to greet Dr. Thorpe, and to extend to him the hand of friendship. They looked forward to an entirely amicable relation between public analysts and the Somerset House authorities under the *régime* of Dr. Thorpe, at whose service the experience of all public analysts would most gladly be placed. Nothing better was desired for public analysts than a close friendship with the Department which Dr. Thorpe represented. This friendship must be based, not only upon a unity of professional interests, but also upon a common desire to be of use to the country, and a mutual wish of scientific men to work hand-in-hand with one another.

Mr. T. H. Elliott said that he rose with the greatest pleasure to respond to the toast which had been so generously proposed by Mr. Hehner. He felt obliged to confess that when he received the invitation with which he had been favoured, he felt some hesitation as to whether he was justified, after so short an interval, in again accepting the hospitality of the Society. But when he heard that Dr. Dyer, a distinguished agricultural chemist, would occupy the position of president, he could not possibly resist the pleasure of supporting him in the chair, and of again meeting the members of their Society. The Board of Agriculture had heard of Dr. Dyer's elevation to the chair of the Society with very special pleasure, on account of his intimate association with agricultural interests during so many years. With reference to the toast which had been proposed, he could not but feel that there was a great community of interest between the Government Departments and the public analysts. They were both the servants of the public; they worked under very similar conditions, and he felt, from what he had read, that the atmosphere of criticism in which Government Departments lived was also freely breathed by public analysts. Reference had been made to chemistry in connection with agriculture. Chemistry had revolutionized the practice of agriculture in this country, and he thought that no agriculturist could look a chemist in the face without a feeling of gratitude for the great benefits which agriculture had derived from that science. Analytical chemistry was of especial interest to agriculturists, and in this connection he was glad to say that the number of analyses made under the Fertilizers and Feeding Stuffs Act was steadily increasing. In the first year during which the Act had been in operation 444 analyses were made; in the next year 615; and in the three-quarters of the year 1896 for which returns had been obtained, the figures of 1895 had already been beaten with 673 analyses; so that the services of public analysts had been called upon more frequently in 1896 than in any year since the passing of the Act.

Agriculturists, too, were not only consumers but also producers. Mr. Kearley had already referred to the special interest which the proceedings of the Food Products Adulteration Committee possessed for agriculturists, who were strongly

represented on that committee, and the Board of Agriculture, and agriculturists generally, owed a debt of gratitude not only to the committee, but to the witnesses who gave evidence before it. He had no authority to speak with regard to future legislation, but he entirely agreed with the view that it would be very desirable that they should have one Act in a convenient and easy form, and he hoped that the passing of such an Act would be shortly followed by the publication of a similar text-book to that for which Dr. Dyer was responsible when the Fertilizers and Feeding Stuffs Act was passed. Both as consumers of manures and feeding-stuffs, and as producers of agricultural products, agriculturists appreciated the services of Public Analysts, and they regarded the Society with the most friendly feelings, because, by the contact of thought with thought which was rendered possible by means of an institution such as theirs, an increase of scientific knowledge and skill was secured. Agriculturists wished the Society every prosperity, believing that such prosperity would be to their mutual advantage, and he was glad to have this opportunity of expressing his gratitude to the Society, and of most heartily wishing it every success in its labours.

Professor THORPE, F.R.S., hoped he would not be misunderstood when he said that he rose with considerable reluctance to return thanks on behalf of the Government Department with which he was connected. His reluctance sprang from the memory of the circumstance that scarcely twelve hours had elapsed since he had had an amicable passage of arms with the President, in which he had been worsted as to the propriety or necessity of what he might term "double-barrelled" speeches. It seemed to him (Professor Thorpe) that when a gentleman so eminently capable as Mr. Elliott of responding for the honour done came forward to perform that duty, nothing more remained to be said. He had a theory, in which he thought he was not singular, that public officials should be like the proverbial good little boy—they might be seen, but should not be too much in evidence. Still, in some respects he might perhaps lay claim to a certain prescriptive right to a share in the response for the various Government Departments, because there were few officials who came into contact with so many of these Departments as he himself did. Indeed, he felt himself, to paraphrase the well-known saying, to be the servant of all and the master of none. The Government Laboratory not only served the Inland Revenue and Customs Departments, but the Board of Agriculture, the Board of Trade, and the Home Office, the War Department, the Admiralty, the India Office, the Local Government Board, and the Post-Office—in fact, he ventured to think there were few Government Departments that did not in some way or other call upon the services of the Government analysts.

Some allusion had been made to a matter in which he naturally took a considerable amount of interest, viz., the work of the committee which had just reported, and from whose endeavours so much was naturally expected. He had read with considerable interest and with very great profit the exhaustive statements contained in the evidence which was brought before the committee, and he might say in a word that with the general conclusions of that report, and especially that section of it which might be considered to affect himself more than any other section, he was entirely and wholly in agreement. The suggested Consultative Board, or Board of

Reference, would be, to any man charged with the duties and functions which he (the speaker) had to perform, a very great boon. The old Adulteration Act was of course a thing of compromises, and it bore upon its very face the evils of compromises. In the administration of that Act a great many difficulties arose which had to be solved in the best way that was open, and in a way which, it must be admitted, when all was said and done, could not be wholly satisfactory. With regard to the question of standards, he might say that at the Government Laboratory they had no legal power whatever to fix standards. The question of standards was a difficulty which was of course constantly arising. Now, the Board of Reference which was spoken of might, he hoped, not only solve this question, which was an extremely knotty one, but many others of interest and importance both to the public analysts and to the Government analysts. Mr. Hehner had more especially alluded to him (Professor Thorpe), and had been good enough to extend, in his characteristic manner, what might be called the right hand of friendship to himself personally. He had already said, upon a previous occasion of a similar nature, and he repeated it, that it was his earnest desire that those at the Government Laboratory should work in perfect harmony with this Society, as representing the great body of public analysts. How this object might best be accomplished was of course a matter to some extent to be determined by the operation of the Bill that was in prospect; but he could not help thinking that those little points of difference (which after all were perhaps not of a very grave nature) would be materially smoothed away by that personal association and friendly discussion which the proposed Board of Reference would inevitably bring about; and therefore, speaking for himself, and he thought he might add for those also with whom he was associated in the work of the Government Laboratory, he agreed most cordially with the suggestion that such a Board should be appointed.

Dr. J. A. Voelcker, in proposing the toast of "The Learned and Professional Societies," said that his mind had been exercised during dinner as to what distinction might properly be drawn between "learned" societies and "professional" societies, but with the help of a neighbour whom he would not specify, he had been able to apply a definition which fairly commended itself to him. It was that a learned society was a society which existed for the advancement of knowledge, while a professional society was a society which existed for the advancement of the individual. Both of these two branches he wished to couple together in the toast which had been allotted to him. Although the Society of Public Analysts was perhaps not so much a learned as a professional society, it must be felt by public analysts that as their work became more and more important, so also it became more and more important that the true principles of science should be thoroughly grounded in them. They would welcome, therefore, the presence among them of Professor Vernon Harcourt, F.R.S., the able representative of the Chemical Society, whose work in a scientific direction was particularly distinguished. They had also the honour of meeting Dr. W. J. Russell, F.R.S., the President of the Institute of Chemistry, and he (the speaker) was sure that public analysts especially would appreciate the efforts made by that Institute, not only under its present President, but also under those who had gone before him, to give to the chemical profession a

proper standing in the eyes of the public. It was a very pleasing reflection that many members of the Society of Public Analysts were also members of these two societies, and of others which were represented on that occasion, namely, the Pharmaceutical Society, the Institute of Brewing, and the Society of Chemical Industry, etc. As to their own Society, although, as he had said, it was perhaps not a learned Society in the stricter sense of the term, still he thought it might claim to be progressing in its learning. It numbered among its members members of some of the most distinguished societies which existed for the advancement of science, and he hoped that its work in aiming at strengthening the public analyst's position and enabling him to grasp and retain the confidence of the public, would be successful and would meet with general approval.

Before concluding, he would like to remind the members of the Society of the fact that, by the kindness of the President of the Chemical Society and his predecessors, their meetings had been allowed to take place in the Chemical Society's Rooms for the last two-and-twenty years; a similar recognition was also due to the President of the Institute of Chemistry, the rooms of that Institute having also been placed at the disposal of the Society upon several occasions.

Mr. A. G. Vernon Harcourt replied, remarking that he could hardly agree with Dr. Voelcker in the distinction he had drawn between a learned and a professional society. There were men who studied chemistry purely in the interests of science, and there were busier—and perhaps more useful—men, whose work lay in the direction of the practical applications of science.

In a pleasant volume of stories written by Mr. Andrew Lang, there was one which bore the title of "The First Radical." The principal character was named—in accordance with the fondness of savages for repetition—"Why-Why." He was a gentleman who never took things as he found them, but always asked for causes. He seemed, in fact, to be a sort of incarnation of the scientific spirit. It might, perhaps, be doubted whether the scientific spirit had not gone too far in this direction, and whether men were not sometimes too anxious to arrive at causes (even to guess them if they could not find them out) rather than to content themselves with the humbler search after facts. But this impatience, this desire for increased knowledge of Nature, the impulse to observe a new fact, to look into it, to inquire "What is it?" "Will it happen again?" "What is the cause of it?" that really was the basis of all scientific inquiry; and its value as a part of human nature was inestimable. This spirit of investigation, for which we had no better name than curiosity or inquisitiveness, this sort of instinct which existed, by no means, he was afraid, in all men, but certainly in many—probably in all who were present on that occasion—and also, he thought, in all children; this impulse to try and find out about things, had been of much service to humanity. The increase of knowledge to which it had led had benefited humanity, for it was the necessary precursor of those technical applications of science of which the utility was obvious. No doubt a good deal of the public support which science received depended upon a well-founded impression that science, somehow or other, however uninteresting it might in itself appear, was of benefit to mankind.

There was another aspect of this question, namely, the change of opinion as to

the universe around us, which had been brought about by increased scientific knowledge. The relation of this world to other worlds, the relation of men themselves to other men and to all other living things, had been cleared up by the investigations which had sprung from this desire for knowledge; and if they compared themselves, as perhaps with some just complacency they might, with at any rate some of the previous generations, it might be justly claimed that to this different way of looking upon the world were due the advances that had been made.

One of the chapters in a well-known book by G. H. Lewes, "The Physiology of Common Life," began with a panegyric on hunger. It was the chief motive power of humanity. Men engaged in their pursuits put out what energy they did put out because of this imperious craving. It seemed to him that those were especially happy in whom this general necessity of nature—the need of earning the means of subsistence—could be satisfied by satisfying at the same time the other craving for fuller knowledge. All of them—whether members of learned societies who were engaged in teaching, or those who, having a knowledge of science, applied their knowledge in dealing with legal questions or as public analysts—all had the advantage of satisfying at the same time these two desires, the desire to know, and the desire for the means of living.

He could not quite agree with his friend Dr. Thorpe in his expression of regret that there should be "double-barrelled" speeches. There were many topics to which he might have referred, but which he was glad to be able to leave in the hands of Dr. Russell. He had spoken of the origin and nature of the scientific spirit, and the advantages it conferred upon those in whom it existed, and upon humanity generally; but he had said very little about the learned and professional societies. The reason why men gathered themselves together—whether to hear papers read and to discuss them, or upon such pleasant occasions as that particular one—was perhaps some gregarious instinct; but there was one very practical and obvious reason for their so doing, and that was the advantage to be gained by combination. It was perhaps fortunate for the Chemical Society that it very rarely needed to approach any Government Department, but in another learned society with which he was connected, viz., the British Association, appeals on behalf of science were made from time to time, and the advantage of combination at once became apparent. He thanked them on behalf of the Learned and Professional Societies.

Dr. W. J. Russell also responded, remarking that chemistry could probably claim, and indeed required, a larger number of really active societies than any other branch of science. The work which these societies had to do rendered it necessary that they should stand shoulder to shoulder in order to carry it on, and all small jealousies between them ought absolutely to be (and he believed at the present time were) annihilated. In expressing his sense of the honour done him in asking him to respond to this toast, he could only say that he believed the Society which he then represented was most honestly and fully deserving of all honour. It did its best to maintain a high professional standard, and admitted into its body only those who could prove they had undergone a lengthened course of study, such as to fit them to be chemical advisers.

Dr. Armstrong, in proposing "The Society of Public Analysts," said that those

who were present had been brought so intimately into connection with the Society as not to require much information concerning it. He personally had long taken a great interest in its proceedings, for shortly before it commenced work he came into office as one of the Secretaries of the Chemical Society, and in that capacity had had every opportunity for taking notice of what the Society of Public Analysts had done. It had now attained its majority, and full information regarding its history up to that point was contained in the admirable presidential address which Dr. Stevenson had delivered to its members some twelve months since. When one looked at the progress that had been made in chemistry during those years, the increase in our knowledge appeared marvellous, and altogether out of proportion to anything attained in any previous period; but he thought he was justified, not only in saying that the progress of the Society of Public Analysts had been entirely proportional to the progress made in the science of chemistry, but also in claiming for the Society a very high position among kindred institutions throughout the world. The public analysts had rendered very valuable service in connection with milk, an article specially important from the fact that young children were fed upon it during the earliest years of their lives; but he did not feel altogether happy when he heard standards mentioned, and was very glad to know from Professor Thorpe that his department, at all events, had no fixed standard; a fixed standard undoubtedly had the effect of raising the quality in some cases, but in a far larger number it resulted in diminution of quality. Only a short time since he had seen an article in the Journal of the Pasteur Institute, arguing that there should be no fixed standard in the case of an article of food like milk. There was another point, namely, the use of preservatives. It was not likely that they could get into the stomach without producing some effect, and the growing use of formalin and of boracic acid, especially the latter, ought to be stamped upon by public analysts. Even if the Society's work had been confined to the subject of milk, its existence would have been justified—but it had done an immense deal more than this. It was very important that they should not confine themselves merely to the carrying out of the technical details of food and drug analysis, and this had been fully recognised by the leaders of the Society. Men like Allen, Dupré, Dyer, Hehner, and others, had shown that the body was not merely engaged in professional duties, but was also occupied in advancing the science of chemistry. Mr. Allen had done immense service by his wonderful book on "Commercial Organic Analysis." Dr. Dupré and Mr. Hehner had given to the world most valuable methods of analysis; and he was sure that the Secretary of the Board of Agriculture would be interested in knowing of the method for measuring the fertility of soils which had been devised by the new President of the Society, Dr. Dyer. With the toast he was proposing he desired to couple the name of Dr. Stevenson, who was a very honoured member of the Society. Dr. Stevenson and he had had occasion to work together for many years in the Chemical Society, and he could say, from his own experience and from that of all his colleagues in the Chemical Society, that Dr. Stevenson was a man worthy of the utmost respect in every way. His dignity of manner, his sobriety of judgment, were well known, and he had done yeoman service on behalf of the Society of Public Analysts during the two years he had been its President. It therefore afforded him (the speaker) great pleasure to couple Dr. Stevenson's name with this toast.

Dr. Stevenson said it was with a feeling of pleasure that he rose to respond to this toast, and he had especially to return his grateful thanks to his old friend Dr. Armstrong for the warm terms in which he himself had been referred to. He had been anxiously looking out for any adverse points which might arise in Dr. Armstrong's remarks with regard to the Society of Public Analysts, for it was well known that Dr. Armstrong, although a warm friend of all science, was yet one of the keenest critics of analytical chemistry; but there were no such adverse points, and this would be esteemed a great honour by anyone who knew Dr. Armstrong.

He (Dr. Stevenson) was one of the founders of the Society, and was its earliest Treasurer. Looking back upon the period of two and twenty years that had elapsed, he felt that the Society was to be congratulated on the advances it had made. Like all progressive societies, it had undergone a good deal of adverse criticism, but, nevertheless, it now comprised most of the important analytical chemists in the kingdom.

Mr. Cassal said that the task which he now rose to perform was an easy and most pleasant one, since it was the proposal of the health of the guests who had honoured the Society with their presence on that occasion. The list was a long one. Dr. Thorpe, the head of the laboratory at Somerset House, they received with the same cordiality that they had ventured to extend to him last year. And they did so not without the hope that in the future the atmosphere that surrounded Public Analysts and the Analysts at Somerset House might be less heavy with thunder-clouds and less seamed with lightning-flashes than it had been in the past. Mr. Elliott, the Secretary of the Board of Agriculture, was also present, and had told them (if not in actual words, at least in spirit) that in the future the working of the Fertilisers and Feeding Stuffs Act would result in the awarding of princely emoluments to those who had the honour to be District Agricultural Analysts. Then they had with them the President of the Chemical Society and of the Institute of Chemistry, and Mr. Fletcher Moulton, with whose name he would couple the toast, and with whom many of them were well acquainted. He believed that in the ante-room that evening either Mr. Moulton or Mr. Ricketts, who was also one of their honoured guests, had been asked the reason of his presence at a gathering of Public Analysts. He thought that all those who had been in a witness-box before Mr. Moulton or Mr. Ricketts would know that the presence of both those gentlemen among Public Analysts was singularly appropriate. He could testify that they were both masters of the art of analysing analysts. The President of the Pharmaceutical Society was also present; the Chairman of the London Section of the Society of Chemical Industry (Dr. Messel); the President of the Institute of Brewing (Mr. A. C. Tanqueray); Mr. C. E. Groves, the valued Editor of the Journal of the Chemical Society; Mr. J. Innes Rogers, a distinguished member of the London Chamber of Commerce; Mr. Beck, a gentleman whom some of the members had met in connection with the settlement of various knotty questions; and many other visitors, in regard to whom he might say that they were all in every sense worthy visitors of the Society of Public Analysts. He asked the members of the Society to drink to the health of those who had honoured them by their presence, and by their

way of doing so to assure them that the Society of Public Analysts received them in all cordiality and with the greatest pleasure.

Mr. J. Fletcher Moulton, in responding, said that he was somewhat surprised that this task should be accorded to him, but he had come to the conclusion that he had been selected as being probably the most remarkable instance of conversion in favour of the support of public analysis. He had to make the confession that he started in early life with a strong prejudice in favour of adulteration. This might perhaps seem somewhat original, but it must be remembered that the history of every man largely depended upon the circumstances of his childhood, and one of the earliest things he could recollect himself was wandering into the laundry and discovering there some poisonous compound which was used in those regions, and which, with that generalizing faculty so characteristic of children, he immediately applied to the purposes of food. The doctor, when sent for, was somewhat astonished to find that the expected symptoms of poisoning had not set in with sufficient vigour, which he (Mr. Moulton) had always attributed to the fact that, even at that early age, his own radical ignorance of the manner in which chemical substances ought to behave had shown itself, and the substance, feeling itself out of place, had not taken the trouble to behave as it should; but his comforted parents were told by the doctor that, providentially, it was a highly-adulterated article. Under these circumstances it was not unnatural that he should start in life with a prejudice in favour of adulteration. It had been his fate, not once or twice, but often, to be accused of appearing in defence of such atrocious practices. The last occasion of this kind that he could remember was in 1881, when he was before a Parliamentary Committee on the subject of the electric light, and had put Sir William Siemens into the box, and had examined him. A very worthy counsel—the representative of the united gas companies—got up and said in a tone of deep solemnity: "Dr. Siemens, electricity is but a young science. Now, Dr. Siemens, will you tell the Committee whether we have got far enough to protect the public against adulteration?" Dr. Siemens had the wit to say that the only instance of adulteration of electricity he had ever heard of was in connection with greased lightning! (loud laughter). He desired, however, having now forgotten the lessons of his childhood, to wish, on his own behalf and on that of his fellow-guests, increased prosperity to the Public Analysts.

Mr. W. F. Keating Stock proposed "The Editorial Committee of the Analyst," coupling with the toast the names of Dr. Sykes and the past presidents of the Society. Gratitude might, he thought, be described as of two kinds, "ancient" and "modern"; "ancient" gratitude being taken to represent a feeling of thankfulness for benefits already received, and "modern" gratitude a lively conception of benefits to come. Both kinds of gratitude would be felt by members of that Society in connection with the duties of the Editorial Committee, and of Dr. Sykes, who had devoted a very large amount of time and labour to the duties devolving upon him as editor.

The toast was briefly acknowledged by Mr. Allen, in the absence of Dr. Sykes.

Mr. Bertram Blount desired to submit a toast which, although not officially announced, would, he was sure, meet with cordial approval. It was the health of those permanent officials who did so much for the Society, though in such an unobtrusive way that their existence was sometimes in danger of being forgotten.

It was well known that the business of no body could go on smoothly without a great deal of work, which must be voluntary, and which might be dull. In submitting the toast of the officials, he would more particularly mention their new President, who for long had done excellent service as Secretary. The work of an official of this kind was not to be computed by the minutes he spent in the official gatherings of the Society; it meant a great deal more than that. It included work performed, very likely, at the expense of his own private occupations, and altogether severed from those cheering influences which surrounded public service. He would like, also, to mention the work of Mr. Bevan, who, in conjunction with Mr. Cassal, the newly-elected secretary, would carry on the secretarial business of the Society; and also their most admirable treasurer, Mr. E. W. Voelcker, who, as they knew, had been seriously ill, and was even now only partially recovered. They were happy to know, as he (Mr. Blount) had learned that evening, that Mr. Voelcker's recovery was going on as well as could be wished, and would, it was to be trusted, be rapid and permanent.

Dr. DYER, in rising to respond, said that there were good reasons why his acknowledgment of this toast should be a brief one. He had that day forfeited the right to reply for the Secretaries, and he had not yet earned the right to speak in the capacity of President. But he would not like to sit down without thanking the members of the Society, one and all, most heartily and most sincerely, for the honour they had done him in unanimously electing him to that chair; and he could only hope that when his term should have expired, he would have done one half of what their late good President, Dr. Stevenson, had done to earn the thanks of that Society.

ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

FOOD AND DRUGS ANALYSIS.

The Critical Temperature of Dissolution. Application to the Analysis of Butter. L. Crismer. (*Bull. de l'Assoc. belge*, 1896, x., 312-316.)—In his several communications on the critical temperatures of dissolution (*cf.* ANALYST, xx., 209 and 257; and xxi., 241), the author has made the determinations in sealed tubes. It is now shown that when the critical temperature does not exceed 78° C., the boiling-point of alcohol, an open tube may be conveniently employed. About 0·5 c.c. of the filtered butter-fat is placed in the reaction-tube (which is 7 to 8 cm. long and about 1 cm. in diameter), with about twice its volume of alcohol, which must be absolute, or nearly so. The tube is closed by a cork, through which passes a thermometer with its bulb entirely covered by the liquid, and not touching the sides of the tube. The tube and thermometer are gently heated in a larger tube, which serves either as an air- or water-bath, the contents being vertically agitated until they give a homogeneous liquid. They are then allowed to cool, and the temperature at which they become turbid is the critical temperature. The small amount of alcohol evaporating during the process does not materially affect the results.

In the following table the alcohol used had a density of 0·7967 at 15·5° C., and contained therefore 0·9 per cent. of water by weight. The dilute alcohol used for the

parallel determinations in sealed tubes had a density of 0·8195 at 15·5° C. (=8·85 per cent. of water). The figures under *a*, *b*, *c*, etc., show the results obtained on repetition after adding a little more alcohol.

CRITICAL TEMPERATURES.

	With Alcohol 0·7967 Specific Gravity.						With Dilute Alcohol in Sealed Tube.	Difference.
Butter.	*a.*	*b.*	*c.*	*d.*	*e.*	*f.*		
1	54·8	54·8	54·8	54·6	54·3	53·8	100·5	45·7
2	54·5	54·5	54·5	54·2	—	—	100·5	46
3	57	57	57	—	—	—	103	46
4	54	54	—	—	—	—	100	46
5	50	50	—	—	—	—	95·5	45·5
6	56	—	—	—	—	—	102	46
7	56·5	56·2	—	—	—	—	102·5	46
8	52	52·4	52·5	52·4	51·2	—	98·2	45·7
Margarine & mixtures.								
1	78	78	—	—	—	—	124	46
2	72·2	72·2	—	—	—	—	118	45·8
3	72·5	—	—	—	—	—	118	45·5
4	78	—	—	—	—	—	123·8	45·8
5	69	—	—	—	—	—	115	46
6	63·8	—	—	—	—	—	109	45·2

When figures are obtained materially lower than those given here for butter, the author's experience is that they are either old and rancid samples, or fresh butters badly prepared from acidified cream. As was pointed out in a former paper (see ANALYST, xxi., 241-243), the lowering of the critical temperature is proportional to the acidity of a fat. By neutralizing such butters with a 10 per cent. solution of potassium carbonate, and washing the fat with hot water, the normal critical temperature is obtained.

It is a curious fact that the number of c.c.'s of alcoholic $\frac{N}{10}$ KOH required to neutralize 2 c.c. of butter fat in 20 c.c. of absolute alcohol, when added to the figure representing the critical temperature of the acid butter, gives approximately the critical temperature of the neutralized butter.

ALCOHOL 0·8195 SPECIFIC GRAVITY AT 15·5° C. IN SEALED TUBES.

Number of Samples.	Mean Critical Temperature. ° C.	Acidity in KOH $\frac{N}{10}$ for 2 c.c. c.c.	Critical Temperature of Neutralized Butter. ° C.	Difference between the two temperatures. ° C.
(*a*) 3 at 80°—90°	88·5	7·5	96·4	7·9
(*b*) 14 at 90°—96°	93·5	4·8	98·2	4·7
(*c*) 69 at 96°—102°	99·12	1·2	101·7	2·6
(*d*) 17 at 102°—106°	103·9	1·0	——	—

See also A. von Asbóth on this subject (ANALYST, this volume, page 21).

C. A. M.

ORGANIC ANALYSIS.

On the Volumetric Determination of Starch by Means of Iodine Solution. Fannie T. Littleton. (*Amer. Chem. Jour.*, 1897, xix., 44-49.)—The object of this investigation was to determine whether small quantities of starch could be determined colorimetrically by means of the iodine reaction, as has been stated by several workers in this direction.

A standard solution of iodine was prepared, which contained iodine dissolved in potassium iodide in the proportion of 1 molecule to 1 molecule, and this was used in conjunction with a standard solution of wheat starch containing 0·1 gramme per litre.

Temperature was found not to have much effect when the rise was not more than 5° C., but above this the colour gradually became fainter, and almost disappeared when the temperature had risen to 15 degrees.

Potassium hydrate, when added to the extent of 0·002 gramme, caused 1 gramme of starch to dissolve rapidly, and did not affect the colour of the starch iodide solution if the alkali were exactly neutralized before the addition of iodine.

Solutions of various kinds of starch were then compared, an exceptionally pure specimen of arrowroot starch being taken as the standard, and the heights of the columns of corresponding colour being compared with this. The strengths (which varied inversely as the heights of the columns) were as follows: Arrowroot, 100; potato, 97·06; wheat, 94·81; tapioca, 90·41; and rice, 83·83. These results were about what one would expect from the known relative size of the starch granules. Hence, rice starch, with its smaller granules and consequently probable larger amount of cellulose, should not show as high a percentage of true starch as arrowroot with its larger granules. Experiments made to determine whether the quantities of starch and iodine required to produce the maximum colour had a definite ratio showed that this was the case. Thus 0·007 gramme of starch required 0·00448 gramme of iodine, and 0·001 gramme required 0·00064 gramme; but this only held good when a solution of iodine of a single strength was employed, for when the same quantity of starch solution was tested with iodine solutions of different strengths, the results were quite irregular. This was ascribed to the dissociating effect of the additional water contained in the weaker iodine solutions, and this view was confirmed by other experiments described in the paper. Therefore, in using the method, it will be necessary to compare substances whose percentages of starch do not differ widely.

It is further shown that chloroform will extract iodine from moist starch iodide, although the contrary is stated in Thorpe's "Dictionary of Applied Chemistry," iii., 565.

The general conclusion arrived at is that, on account of the easy dissociation of the so-called starch iodide, the method is only available with substances containing a large amount of starch, and the colorimetric comparisons must then be made under conditions very nearly identical. This limits very considerably the application of the process in the examination of food material, although in occasional cases it may be of value. C. A. M.

On the Purification of Phenyl-Glucosazone. L. Hugounenq. (*Jour. Pharm. Chim.*, 1896, 447, 448.)—Anisol ($C_6H_5.O.CH_3$) is substituted for acetone in the re-crystallization of the washed glucosazone. The impure osazone is washed in the cold with water, alcohol, and acetone, re-crystallized from hot anisol (B.P. 152-154°) and the crystals washed with dilute alcohol. They are then perfectly pure, and melt at 204°-205° C. The advantages claimed are that less solvent is required, and that the penetrating odour of acetone is avoided. C. A. M.

On the Messinger Method of Estimating Acetone. H. C. Geelmuyden. (*Zeit. anal. Chem.*, 1896, xxxv., 503-516.)—Messinger's method (*Zeit. anal. Chem.*, xxix., 696) consists in adding potassium hydrate to the acetone solution, then shaking with an excess of standard iodine solution, liberating the excess of iodine with hydrochloric acid, and titrating with thiosulphate. The author employed this method for the estimation of acetone in the breath and urine of animals, and having to work under very varying conditions, made control experiments to determine to what extent these influenced the accuracy of the results.

For determining the amount of acetone given off in the breath by rabbits a respiration apparatus, constructed on Pettenkofer's principle, was employed. The animals were enclosed in a cage through which a continual stream of air was drawn, which, on issuing from the cage, was divided into a main and branch current, both of which were mechanically measured. It was not possible to determine the acetone in the branch current by conducting this through a mixture of potash and iodine solution, since, as was shown by Collischonn, the mixture soon ceased to convert acetone into iodoform, and after half an hour became completely useless. But by passing the current through strong potash alone, all the carbon dioxide and a portion of the acetone were retained, which latter could be subsequently titrated. The air was then passed through a tube containing hot copper oxide, and the carbon dioxide produced was absorbed by baryta in a modification of Pettenkofer's tube (ANALYST, this volume p. 83).

Blank experiments proved that besides acetone no other volatile organic substance was given off in the breath of animals; and in other experiments in which weighed quantities of acetone were volatilized in the empty cage the amount found corresponded with that taken within from 5 to 10 per cent.

It was also proved that the strong potash required by this method did not have any decomposing influence on the acetone retained by it (at least within twenty-four hours), but before adding the iodine solution it was found necessary to dilute the liquid with from 2 to 3 times its volume of water, since, otherwise, the formation of the iodoform was so instantaneous that particles of iodine were mechanically enclosed.

In the Messinger-Huppert method of estimating acetone in urine a certain amount of loss appears to be inevitable, this loss, according to the author's experience, increasing with the amount of acetone present. In experiments in which only trifling amounts of acetone were added, the mean loss in five determinations was 6·75 per cent. of the total quantity added when the distillate was cooled with ice, and 8·30 per cent. when this precaution was not taken. Before adding the iodine solution

to the acetone distillate, the latter, when cooled with ice, should be raised to a temperature of 18° to 20° C. by the addition of hot water.

Since all normal urine contains traces of volatile substances which combine with iodine, a correction must be made for these. In the case of human urine this amounted to from 0·58 to 1·35 milligrammes, calculated as acetone per 100 c.c.

The addition of urea is not advisable, unless the urine under examination contains large quantities of nitrites, since the distillation, even without the addition, yields results which are too low, and the urea usually lowers the amount still further.

C. A. M.

A Contribution to the Chemistry of Animal Fats. C. Amthor and J. Zink. (*Zeit. anal. Chem.*, 1897, xxxvi., 1-17.)—While introducing no new methods, the authors have determined all the usual constants of the fat of a large number of animals and birds which, in many cases, had never been previously examined. Their mean results are given in the subjoined table, in which the Hübl number was determined by adding an excess of 50 to 60 per cent. of iodine, the Reichert number on 2·5 grammes of fat, and the acetyl number by the original method of Benedikt. The melting-point was taken in a capillary tube, closed at one end, and the two numbers given are respectively those of the commencement and end of the fusion.

Fat.	Sp. Gr. at 15° C.		Melting Point.		Solidification Point.		Iodine No.		Saponification No.		Hehner No.	Reichert No.	Acetyl No.	Acidity No.	
	Fat.	Fatty Acids.	Fat, °C.	Fatty Acids, °C.	Fat, °C.	Fatty Acids, °C.	Fat.	Fatty Acids.	Fat.	Fatty Acids.				Fresh.	Old.
Elk ..	0·9625	0·9584	49-52	53-55	37-38	48-50	35·0	27·8	195·1	201·4	—	0·78	16·2	0·87	3·3 (2 years old)
Stag ..	0·9670	0·9685	51-52	50-52	39-40	46-48	25·7	23·6	199·9	201·3	—	1·66	16·4	3·50	5·9 (1 year)
Fallow Deer	0·9615	0·9524	52-53	50-53	40	47-48	26·4	28·2	195·6	201·4	—	1·70	18·4	2·90	5.3 (1 year)
Chamois	0·9697	0·9546	54-56	57-58	42-43	51-52	25·0	24·4	203·8	206·5	—	1·80	7·5	3·20	—
Roebuck	0·9659	0·9622	52-54	62-64	39-41	49-50	32·1	27·9	199·0	200·5	95·8	0·99	12·0	1·74	3·3(some months)
Wild Boar	0·9424	0·9333	40-44	39-40	22-23	32·5-33·5	76·6	81·2	195·1	203·6	—	0·68	29·3	2·60	4·5 (1½ years)
Dog ..	0·9229	0·9278	37·5-40	39-40·5	21-23	34·5-35·5	58·5	50·15	195·4	199·15	95·65	0·57	10·9	1·79	3 (2 years)
Fox ..	0·9412	0·9492	35-40	41-43	24-26	36-37	79·7	65·4	191·7	205·7	—	1·30	43·1	5·90	15·9 (2 years)
Badger ..	0·9226	0·9230	30-35	34-36	17-19	28-30	71·3	73·0	193·1	193·7	96·0	0·86	13·1	5·30	7·2 (1½ years)
Domestic Cat	0·9304	0·9251	39-40	40-41	24-26	35-36	54·5	54·8	190·7	—	96·0	0·90	10·0	2·30	25·6 (1 year)
Wild Cat	0·9304	0·9366	37-38	40-41	26-27	36-37	57·8	58·8	199·9	203·8	—	2·5	19·5	9·30	—
Pine Marten	0·9345	—	33-40	39-43	24-27	35-37	70·2	58·0	204·0	—	93·0	1·10	—	12·65	—
Polecat ..	—	—	—	34-40	—	36-27	62·8	60·6	—	—	—	—	—	—	—
Hare ..	0·9349	0·9361	35-40	44-47	17-23	36-40	102·2	93·3	200·9	209·0	95·2	1·59	34·8	2·73	8 (6 months)
Tame Rabbit	0·9342	0·9264	40-42	44-46	22-24	37-39	69·6	64·4	202·6	218·1	—	2·80	81·0	6·20	—
Wild Rabbit	0·9393	0·9420	35-38	39-41	17-22	35-36	99·8	101·1	199·3	209·5	—	0·70	41·7	7·20	—
Goose ..	0·9274	0·9257	32-34	38-40	18-20	31-32	67·6	65·3	193·1	202·4	—	0·98	27·0	0·59	—
Wild Goose	—	—	—	34-40	—	33-34	99·6	—	—	—	—	—	—	—	—
Wild Goose*	0·9158	0·9251	—	36-38	18-20	32	67·0	65·1	196·0	196·4	—	—	41·6	0·86	—
Duck ..	—	—	36-39	—	22-24	—	58·5	—	—	—	—	—	—	—	—
Wild Duck	—	—	—	36-40	15-20	30-31	84·6	—	198·5	—	—	1·30	—	—	—
Turkey ..	0·9241	0·9283	33-40	38-40	21-27	32-34	66·7	64·6	193·5	200·8	—	1·00	—	1·50	2·3(some months)
	0·9220	0·9385	—	38-39	—	31-32	81·15	70·7	200·5	210·1	—	—	—	4·00	—
Blackcock	0·9296	0·9374	—	30-38	—	25-28	121·1	120·0	201·6	199·3	—	2·10	45·3	5·90	—
Pigeon ..	—	—	—	38-39	—	33-34	82·3	—	—	—	—	—	—	—	—
Starling..	—	—	30-35	38-39	15-18	30-31	83·7	79-4	209·2	—	—	—	—	—	—

* Two years in captivity.

Referring to the above results, the authors drew a number of general conclusions: In the case of tallow-like fats a high specific gravity of the fat was accompanied by a high melting-point and low iodine number. The iodine number of the fats and fatty

acids decreased on keeping, whilst, on the other hand, in the case of many fats, such as that of the stag, dog, and wild boar, the acidity number increased. The acetyl number of the fats also became greater with age. The iodine number, and generally the acetyl number, were lower in the fat of domestic animals than in that of the corresponding wild animals. Similarly, a difference was noticeable in the case of the birds, the fat of the domestic goose, hen, and duck being lard-like, whilst that of the related wild birds was oily.

In four cases the fats exhibited marked drying properties, three of them becoming quite solid in the course of seven or eight to twelve days when spread in a thin layer on a glass plate. This property was possessed by the fat of the hare, wild rabbit, wild boar, and, in a lesser degree, by that of the blackcock.

The fat of the wild boar differed from common lard in having a higher specific gravity, iodine number, and acetyl number, and especially in the above-mentioned drying property.

Fox-fat differed from that of the cat and dog in its higher iodine number and specific gravity, but more particularly in its high acetyl number; while the principal differences in the fats of the common and wild cats were the higher Reichert and acetyl numbers, and the considerably higher acidity numbers of the latter.

Polecat-fat was quite liquid, and showed somewhat lower constants than the fat of the marten. The fat of the dog and cat were very similar in appearance to lard, which they also resembled in analytical results, with the exception of the acetyl value.

Of the different bird-fats examined, that of the blackcock was noticeable for its drying properties and high iodine number. When spread in a layer on glass it gradually set to a varnish, which, however, still remained sticky. After fourteen days drying was still proceeding. After being dried for ninety-five days, the iodine number of the fat had fallen to 29·7. C. A. M.

The Separation of Alkaloidal Extracts. C. Platt. (*Jour. Amer. Chem. Soc.*, 1896, xviii., 1104.)—The persistent emulsions which frequently occur in the analysis of alkaloids, as, for instance, in the ether and benzene extractions by the Dragendorff method, may be separated in a few minutes by filtration through cotton-wool in the following manner: The filter tube, which is similar to a Soxhlet tube, is fitted through the cork of an Erlenmeyer flask, while a stout platinum wire, bent at the upper end, passes through the constricted portion of the tube to the bottom of the flask. Washed cotton is packed firmly in the tube to a depth of about 4 c.m., and the apparatus is connected with a filter-pump. After filtration the separated liquids may be carefully poured into a separating-funnel and treated in the usual way.

C. A. M.

Estimation of Caffeine in Tea, Coffee, etc. A. Delacour. (*Journ. Pharm. Chim.*, 1896, 490, 491.)—It is claimed that the following method is very rapid, and gives completely accurate results: Two grammes of the finely-divided substance are boiled in a 100 c.c. flask for 10 minutes with 80-90 c.c. of water; after cooling, 4 c.c. of basic lead acetate are added, the liquid made up to the mark, shaken and filtered.

The alkaloid contained in 50 c.c. of the filtrate is then extracted with chloroform in the usual manner and weighed. In the case of green tea or green coffee the caffeine is crystalline and colourless, but with roasted coffee it is slightly brown.

C. A. M.

The Assay of Fluid Extract of Coca. C. T. Kingsley. (*Amer. Jour. Pharm.*, 1896, lxviii., 609.)—Eight fluid extracts representative of those found in the market in the United States were examined. After preliminary experiments, Lloyd's process was found to give the best results. The green coloring matter was removed by dissolving the crude alkaloid in acidulated water and filtering; the solution was then made alkaline with ammonia, and the alkaloid extracted by shaking out with chloroform. In each assay 10 c.c. of the fluid extract were taken. The percentage of cocaine found varied from 0·335 to 0·675, the mean being 0·544. C. A. M.

The Separation of Codeine and Morphine. L. Fouquet. (*Jour. Pharm. Chim.*, 1897, xvii., 49, 50.)—This method of separation depends on the greater solubility of codeine in anisol. Pure anisol (specific gravity 0·991 and B.P. 152°) dissolves codeine readily in the cold, and its solvent power increases as the temperature rises. Morphine, on the other hand, is quite insoluble in cold anisol, and only slightly soluble in the boiling liquid. The following table illustrates this difference in solubility at different temperatures:

Temperature	0° C.	16° C.	32° C.	100° C.	150° C.
Morphine, per cent.	insol.	insol.	insol.	0 95	4·80
Codeine, per cent.	7·80	15·28	—	164·00	—

It is remarkable that the solubility of codeine is greater after it has been crystallized from anisol. Thus, after one crystallization the alkaloid dissolved to the extent of 10·75 per cent. at 0° C.

An experiment was made on a mixture of 1·044 gramme of codeine and 0·710 gramme of morphine. This was exhausted by trituration with 20 c.c. of anisol at 15° C., and the residue washed with an addition of 10 c.c. and dried. The amount of morphine recovered was 0·702 gramme. C. A. M.

Estimation of Nitrogen in Guanos, etc. V. Schenke. (*Chem. Zeit.*, 1896, xx., 1031.)—In consequence of certain criticisms on his past work, the author has reinvestigated three of the different processes that have been recommended for this analysis: (1) Förster's modification of Jodlbauer's process, (2) the Ulsch-Kjeldahl method, and (3) Haselhoff's plan of extracting the guano with water. The samples examined were 10 in number, consisting of natural guanos, mixtures of the latter with increasing amounts of sodium nitrate until they contained about 5 per cent. of nitrogen as N_2O_5, and ordinary commercial saltpetre, the total nitrogen present varying from 0·84 to 15·65 per cent.

The results obtained show that the Ulsch-Kjeldahl process always gives the correct yield of nitrogen; and that the Jodlbauer-Förster method is equally exact,

not only up to the limit of 2 per cent. of nitrate nitrogen, as stated by Franke,* but even when 5 per cent. is present. In two cases Haselhoff's process gave only 6·70 and 6·80 per cent. of total nitrogen, instead of 7·71 and 7·46 per cent. respectively; and further investigation showed that this deficit was due, not to "easily soluble organic nitrogen compounds" (Franke, *loc. cit.*), which expression can only mean ammonium oxalate, but to such substances as uric (or hippuric) acid and guanine. These latter, on distillation with caustic soda or reduction with iron and sulphuric acid (Ulsch), only yield from 2·7 to 11·8 per cent. of their nitrogen in the shape of ammonia.

Haselhoff's extraction process, therefore, gives satisfactory results when uric acid, etc., is practically absent; but it is not adapted for the analysis of guano and similar manures.

In the presence of much nitrous acid, both the Ulsch-Kjeldahl and the Jodlbauer methods return too little total nitrogen; it is consequently necessary in such cases to oxidize the substances first; but the author has not yet succeeded in elaborating a convenient plan for doing this. F. H. L.

INORGANIC ANALYSIS.

The Estimation of Cadmium as Oxide. By Philip E. Browning and Louis C. Jones. (*Zeits. Anorg. Chem.*, xiii., 2 and 3, p. 110.)—The authors have repeated Muspratt's methods for the estimation of cadmium as oxide, and show that the inaccuracies which he found, owing to reduction of the oxide, can be avoided, and the precipitation of the element as carbonate and conversion into oxide serves as a reliable method for the determination of cadmium.

The authors operated on quantities of cadmium sulphate corresponding to from 0·1140 to 0·2555 gramme cadmium oxide. They proceeded in the following manner: Weighed quantities of cadmium sulphate were dissolved in hot water, diluted to about 300 c.c., and a 10 per cent. solution of potassium carbonate was slowly added, and the solution shaken constantly, until no further precipitation took place; the solution was then boiled for fifteen minutes, and the precipitated carbonate converted into the crystalline form. The solution was then filtered into a Gooch crucible containing a layer of ignited asbestos, the precipitate, after careful washing, was dried, and heated to redness until constant. The authors show that the estimation yields reliable results, and that the process is much more expeditious than that proposed by Muspratt. The results are, however, liable to be a little too high, owing to the presence of small quantities of potassium carbonate.

H. C. L. B.

The Quantitative Estimation of Bismuth. By W. Muthmann and F. Mawrow. (*Zeits. Anorg. Chem.*, xiii., 2 and 3, p. 209.)—The authors show that bismuth can be accurately estimated, as such, by precipitation with hypophosphorous acid,

* Franke apparently referred to the original form of the Jodlbauer process (cf. ANALYST, xxi., 164), which the present author objects to on the ground of its being too tedious.—ABS.

and they state that their method in many cases exceeds, in accuracy and speed, those hitherto known. The experiments were carried out with bismuth oxychloride, which was suspended in a little water, and solution effected by the addition of a few drops of hydrochloric acid.

The following procedure is recommended as yielding the best results:

To the somewhat concentrated, but not too strongly acid, solution is added an excess of hypophosphorous acid, and the solution warmed on the water-bath until the supernatant liquid is clear; the solution should then be heated to the boiling-point, and a further addition of the reagent should cause no further coloration. The metal separates as a reddish-gray spongy mass (which is readily filtered and washed); this is collected either upon a weighed filter-paper, or in a Gooch crucible, washed first with boiling-water, and then with absolute alcohol, and dried at 105°.

The filtrate should be tested for bismuth with hydrosulphuric acid, when a slight brown coloration will be obtained if the solution has not been warmed sufficiently long.

The method, it is claimed, serves also for the separation of bismuth from such metals as lime and cadmium, which are not precipitated by hypophosphorous acid.

H. C. L. B.

The Separation of Vanadium from Arsenic. C. Field and E. F. Smith. (*Jour. Amer. Chem. Soc.*, 1896, xviii., 1051, 1052.)—The method is based on the difference in volatility of the sulphides of vanadium and arsenic, when heated in an atmosphere of hydrochloric acid gas, the latter being completely expelled at a temperature little above 150° C., whilst the brown vanadium sulphide is not altered. In the experiments the results of which are given below both sulphides were in perfectly dry condition, and the temperature was not allowed to exceed 250° C.:

Taken.		Left.
Vanadium Sulphide. Gramme.	Arsenic Sulphide. Gramme.	Vanadium Sulphide. Gramme.
0·1303	0·1302	0·1297
0·1290	0·2242	0·1297
0·0828	0·0582	0·0827
0·1306	0·2028	0·1308
0·1403	0·2409	0·1404

The following course was adopted in the analysis of a specimen of vanadinite: 0·2500 gramme of the air-dried and finely-divided mineral was placed in a porcelain boat, which was gently heated in a combustion-tube in a current of dry hydrochloric-acid gas. This treatment expelled vanadic and arsenic oxides, leaving lead phosphate and chloride. The receiver containing the vanadium and arsenic was made alkaline and digested with ammonium sulphide, and from the solution of the sulpho-salts the sulphides were set free by a dilute acid. After washing and drying, the sulphides were separated in the manner indicated above, then converted into oxides, and determined in the usual way. The sum of the constituents determined as lead oxide, phosphoric oxide, vanadic and arsenic oxides, with some lead chloride, amounted to 0·2501 gramme.

C. A. M.

The Separation of Manganese from Copper and Zinc, and of Copper from Zinc and Nickel, etc. P. Jannasch. (*Zeit. f. anorg. Chem.*, xii., 2, 134-142.)—1. Separation of manganese from zinc.—Dissolve about 0·4 gramme of the substance under examination in 10-15 c.c. of water slightly acidulated with hydrochloric acid. Add 15 c.c. of glacial acetic acid, and pour drop by drop, with constant stirring, into a mixture of 40-50 c.c. concentrated ammonia and 30-40 c.c. hydrogen peroxide (3 per cent.). Cover and heat on a water-bath for fifteen minutes. Filter off the precipitate, wash with concentrated and then with dilute ammonia, and finally with warm water. It is then absolutely free from zinc, which latter may be estimated in the filtrate.

2. Manganese from copper.—Proceed precisely as in 1, using, however, a mixture of 60 c.c. ammonia and 50 to 60 c.c. hydrogen peroxide. Heat for half an hour, filter off and wash several times by filling up the filter with a warm solution (30 per cent.) of ammonium acetate in strong ammonia. Then wash with ammonia alone, and finally with hot water.

The copper is estimated in the filtrate by any of the usual methods, or, if preferred, it may be removed from the solution first either (1) by hydrogen sulphide, or (2) by potassium or ammonium sulphocyanide. In the first case the author advocates the use of sulphuric acid, as above, instead of hydrochloric. After filtering off the copper sulphide evaporate the filtrate to get rid of SH_2, and then pour into a mixture of 30 c.c. strong ammonia, and 30 c.c. hydrogen peroxide, and treat as already described. If the copper is precipitated as sulphocyanide, destroy the excess of the precipitate in the filtrate in the usual manner with hydrochloric and nitric acids, and determine the manganese as already described.

The hydrogen peroxide method has yielded excellent results in the author's hands, and its advantages, as compared with methods in common use, are discussed at some length. C. H. C.

The Valuation of Borax. J. G. Heid. (*Zeit. angew. Chem.*, 1896, 679.)—The usual commercial method is to separately determine the various impurities and deduct the sum of these from 100, the difference being taken as the amount of crystallized sodium diborate ($Na_2B_4O_7 + 10H_2O$). Since the amount of various impurities is frequently very great, the analysis often requires a long time, and in consequence the author has devised the following method:

Ten grammes of the borax are dissolved in 250 c.c., and the sodium chloride estimated gravimetrically in an aliquot portion of the solution. A second portion (25 c.c.) is diluted with water in a flask, and boiled with an excess of hydrochloric acid for five minutes. The liquid is then evaporated to dryness on the water-bath, and the sodium chloride in the residue determined. From the result that previously found is deducted, and the difference calculated into the equivalent of sodium diborate. C. A. M.

The Use of Boric Acid in the Analysis of Silicates. P. Jannasch. (*Zeit. f. anorg. Chem.*, xii., 3, 208-218.)—The process consists in fusing the mineral with finely powdered boric acid, and subsequently removing the latter by repeated evapora-

tion with methyl chloride. The pure boric acid, free from alkalis, is prepared by recrystallizing the commercial salt, first from 2 to 3 per cent. hydrochloric acid, and then from water. It is dried, fused, and powdered, as previously described (ANALYST, xx. 51).

The methyl chloride must be freshly prepared, as it seems to lose its efficiency after long keeping. For this purpose the author employs a wash-bottle with ground-in tubes, instead of the usual cork, into which he pours 250 c.c. of pure anhydrous methyl alcohol. A rapid current of hydrochloric acid gas is then passed through the cooled liquid for two or three hours.

The analysis is conducted as follows:

Mix in a crucible of 60-65 c.c. capacity 1 gramme of the powdered silicate with from three to eight times the quantity of boric acid, the proportion depending on the ease with which the substance is decomposed. Heat gently for five to ten minutes, then strongly, destroying any large bubbles that may be formed with a short platinum wire. When the formation of bubbles has almost stopped, cover the crucible, and continue heating, finally using a blowpipe flame. Place the hot crucible on a pipe-clay triangle immersed in cold water, weighting the lid as a further security against loss. Remove the contents, as far as possible, to a large platinum dish, soak in water, add hydrochloric acid, and evaporate to dryness. Now add about 60 c.c. of methyl chloride, wash down the sides of the dish with the same liquid, and evaporate at a temperature of about 75-80°. Repeat with fresh quantities of methyl chloride three or four times. Wash down the *outside* of the dish (which may have a slight incrustation of boric acid) with methyl chloride into a large clock-glass, and evaporate to dryness—no residue should be left.

The complete removal of the boric acid from the contents of the dish may be ascertained by placing a Bunsen burner near the water-bath, and fanning a little of the vapour towards the flame, which will exhibit the characteristic green colour if any boron compound is still being evolved. A clock-glass should be at hand to place over the dish should the vapour catch fire.

After the final evaporation with methyl chloride, heat the dish for an hour to 110° C., then add 5 c.c. of hydrochloric acid, and proceed in the usual manner.

The method previously described, although of almost universal application for the analysis of silicates is not applicable to the analysis of minerals of the Andalusite group. In the analysis of one of them ("cyanite") the author found treatment with calcium fluoride and hydrogen potassium sulphate, etc., quite unavailing; but, after various experiments, succeeded in effecting complete decomposition by the following means: The powdered material was put in a crucible with 10 c.c. of strong ammonia, diluted with water, and strongly acidulated with hydrofluoric acid, evaporated, fused for some time, the excess of ammonium fluoride being driven off. The silico-fluoride thus produced was treated with dilute sulphuric acid and evaporated, and the excess of acid driven off. The residue was then found to entirely dissolve in dilute hydrochloric acid.

The rest of the paper is occupied with the composition of the mineral in question.

C. H. C.

The Iodometric Determination of Selenious and Selenic Acids. J. F. Norris and H. Fay. (*Amer. Chem. Journ.*, 1893, xviii., 703-706).—It is claimed that the method here described requires fewer reagents, occupies less time, and gives more accurate results than that of Gooch and Peirce (*Am. J. Sci.*, i. [4] 31), hitherto considered the most suitable volumetric process. It depends on the reaction between sodium thiosulphate and selenious acid in the presence of hydrochloric acid, in which, although the complete reaction is not known, one molecule of selenious acid is exactly equivalent to four molecules of sodium thiosulphate.

Determination of Selenious Acid.—The selenious acid used in the test experiments was prepared by evaporating commercial selenium to dryness with concentrated nitric acid, reducing the dioxide thus obtained by sodium sulphite and hydrochloric acid, again oxidizing the selenium with nitric acid and subliming the residue. About two grammes of the selenious acid were dissolved in water and made up to 500 c.c., aliquot portions being taken for each determination.

The definite portion taken was diluted with ice-water and 10 c.c. of hydrochloric acid (1·12 sp. gr.), an excess of decinormal thiosulphate solution added and titrated back with standard iodine solution. The titration was not affected by a large excess of hydrochloric acid when the solution was cold. Results:

SeO_2 taken. gramme.	SeO_2 found. gramme.	SeO_2 taken. gramme.	SeO_2 found. gramme.
0·0829	0·0829	0·1366	0·1367
0·1242	0·1242	0·1656	0·1659
0·1242	0·1242	0·2070	0·2071

Determination of Selenic Acid.—The selenic acid was prepared by dissolving about two grammes of selenium dioxide in 200 c.c. of water, and adding a strong solution of potassium permanganate until a pink colour remained after heating for thirty minutes at 60° C. Sulphurous acid was then added until only a small amount of manganese dioxide remained, and the solution filtered and diluted to 500 c.c.

In making a determination 25 c.c. of concentrated hydrochloric acid were added to a definite portion of this solution, which was then diluted to 100 c.c. After being boiled for an hour, care being taken not to allow the volume to go below 75 c.c., the liquid was cooled, diluted with ice-water, and the selenious acid determined by titration. Results:

SeO_3 taken. gramme.	SeO_3 found. gramme.	SeO_3 taken. gramme.	SeO_3 found. gramme.
0·1011	0·1009	0·1598	0·1595
0·1067	0·1067	0·2023	0·2024
0·1067	0·1065	0·2665	0·2662

Determination of a mixture of Selenious and Selenic Acids.—Two portions of a solution containing known quantities of selenious and selenic acid were measured off. In one the selenious acid was determined by direct titration; in the other the total selenium, after reduction of the selenic acid. Some selenium was precipitated on heating the solution during the reduction, but this dissolved when the chlorine was evolved. Results:

SeO_2 taken. gramme.	SeO_2 found. gramme.	SeO_2 taken. gramme.	SeO_2 found. gramme.
0·0467	0·0467	0·1013	0·1015
0·0467	0·0466	0·1013	0·1016
0·1868	0·1865	0·1012	0·1015

The reagents used in the test experiments were all carefully purified. Resublimed iodine was sublimed from one-third its weight of potassium iodide, and dissolved in potassium iodide free from iodate. The sodium thiosulphate was purified by recrystallization, and the solution standardized with resublimed iodine and pure sodium arsenite. C. A. M.

Alcohol as a Source of Error in Volumetric Analysis. L. F. Kebler. (*Amer. Jour. Pharm.*, 1896, lxviii., 667-673.)—Referring to Caspari's paper (ANALYST, xxi., 293), the author describes experiments which prove the correctness of the conclusions there stated as to the disturbing influence of commercial alcohol in volumetric analysis, but it is further shown that absolutely pure alcohol does not so interfere, except in the case of methyl orange and tropæölin 00. Absolute alcohol was prepared by repeated fractional distillation from commercial alcohol which had been made faintly alkaline with potassium hydrate, and with the purified product thus obtained, the results on titrating alkaloids and alkaloidal residues in alcoholic solution were as accurate as those found in aqueous solution. C. A. M.

APPARATUS.

A New Baryta Tube. H. C. Geelmuyden. (*Zeit. anal. Chem.*, 1896, xxxv., 516, 517.)—In estimating the amount of carbon dioxide in the air by Pettenkofer's method of drawing a known volume of the latter through a tube containing standard baryta water, and titrating an aliquot portion of this with acid after the absorption, it may easily happen that the baryta water changes in volume during the experiment, especially when this is long continued. As a remedy for this, the author has constructed a tube on the principle of the Gay-Lussac burette, which during the absorption is placed almost horizontally, with the open ends of the tubes bent upwards. The wide tube is graduated, and the volume of baryta water is read before and after the absorption, the tube being placed in a vertical position during the readings. C. A. M.

LEGAL.

PROCEEDINGS IN THE COURT OF APPEAL.

MILK OF SULPHUR PROSECUTION.

(*Reprinted from the "Pharmaceutical Journal" of February* 6, 1897.)

The case of Sandys v. Simpson came before Mr. Justice Wright and Mr. Justice Bruce in the Divisional Court, on Monday last, upon a case stated by the Justices of Heanor in the county of Derby. It appeared that at the Petty Sessions held at Heanor on October 26 last,

an information was preferred by Henry Stair Sandys against a chemist and druggist named David Osborne Simpson, for that he (the respondent), did on August 18, 1896, at Heanor, sell to one Joseph Hewitt, to his prejudice, a certain drug, namely, "precipitated sulphur," which was not of the nature, substance and quality of the article demanded by the purchaser, in that it contained 46 per cent. of sulphate of lime. The appellant, Mr. H. S. Sandys, is an inspector of Weights and Measures for the county of Derby, charged with the execution of the Sale of Food and Drugs Act, 1875, under Section 6 of which the information was laid. When the matter was before the Justices, on the charge being read over to the respondent, he did not plead guilty or not guilty, but before any evidence was given, admitted selling the article complained of, namely, milk of sulphur, and stated that when he was weighing it he informed Joseph Hewitt that he had two preparations of the article, viz., "milk of sulphur" and "precipitated sulphur." The respondent offered to change the article, and to supply Hewitt with precipitated sulphur, which he designated as "the pure," but Hewitt insisted on being supplied with the article that the respondent was in the act of weighing, viz., milk of sulphur, and he was accordingly served with milk of sulphur. The respondent further stated that milk of sulphur was a preparation he had sold for forty or fifty years, and that it was what his customers usually expected to be supplied with. He also stated that it was his custom to keep pure precipitated sulphur for medicinal purposes, but that he was seldom asked for it and his customers had repeatedly returned it to him when supplied to them, and asked that they might instead be supplied with milk of sulphur, which was the article he (the respondent) at first thought Hewitt required. The respondent further made the following statement: "I rather sealed my own doom in that I used a printed precipitated sulphur label without writing on it 'milk of sulphur,' but I had used up all the 'milk of sulphur' labels a few weeks ago, and being in a hurry and not very well that day I put this 'precipitated sulphur' label on in mistake." It was proved that Hewitt was the County Inspector and was at Heanor on August 18, 1896. Hewitt, acting on the appellant's directions, that day went to the respondent's shop and asked for half a pound of precipitated sulphur. Hewitt duly notified to the respondent his intention to have the article analysed by the public analyst, and offered to divide it into three parts in accordance with Section 14 of the Statute, and duly divided it into three parts, delivering one part to the respondent. The requirements of the Statute were complied with, and upon one of the parts being analysed by the Public Analyst it was found to contain 46 per cent. of sulphate of lime. The appellant alleged that the article supplied and sold by the respondent to Hewitt was not of the nature, substance, and quality of the article demanded by Hewitt, who on cross-examination denied the allegation that the respondent had offered whilst weighing the milk of sulphur, or at any other time, to supply precipitated sulphur, or to change it. The respondent was not sworn, nor was any evidence called on his behalf. The information was heard before four justices of the peace, and was dismissed on the decision of the majority. The grounds of the majority for dismissing the information were that the respondent was well known to them as a most respectable and honest tradesman, and they fully believed his statement, notwithstanding the evidence given by Hewitt.

Mr. Hextall appeared in support of the appeal.

The respondent was not represented.

Mr. Justice Wright said he should think very likely the Justices were wrong in this case, but they had a right to go wrong if they could reconcile it with their consciences. At any rate that Court could not set them right.

Mr. Justice Bruce concurred.

Appeal dismissed without costs.

THE ANALYST.

APRIL, 1897.

PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS.

THE monthly meeting of the Society was held on Wednesday evening, March 3, at the Chemical Society's Rooms, Burlington House, the President (Dr. Bernard Dyer) being in the chair.

The minutes of the previous meeting were read and confirmed.

The following gentlemen were proposed for election—as members: Leonard de Koningh, F.I.C., Analytical Chemist, 325, Kennington Road, S.E.; Alexander Henry Mitchell Muter, A.I.C., Analytical Chemist, 325, Kennington Road, S.E.; William Crowder, F.I.C., Analytical and Consulting Chemist, 271, Evering Road, Upper Clapton, N.E.; Alexander Edwin Tucker, F.I.C., Analytical Chemist, 35, Paradise Street, Birmingham; John Heron, F.I.C., Analytical and Consulting Chemist, 110, Fenchurch Street, London. E.C. As associate: Richard Murray, assistant to Mr. Benjamin Watmough, Wakefield.

The following papers were read: "The Composition of Milk and Milk Products," by H. Droop Richmond; "The Detection of Mixtures of Diluted, Condensed, or Sterilized Milk with Fresh Milk," by H. Droop Richmond and L. K. Boseley; "The Estimation of Milk-sugar in Milk," by H. Droop Richmond and L. K. Boseley; "The Constitution of Milk," by H. Droop Richmond; "Copper in Preserved Vegetables," by R. Bodmer and C. G. Moor, M.A.; "Coffee Palace Coffee Infusions," by E. G. Clayton.

The following gentlemen were elected—as members: Hugh Candy, B.A., B.Sc., Lecturer on Chemistry at the London Hospital; Frederick William Harris, Public Analyst for Burnley (Lancashire); John H. B. Jenkins, Chemist to the Great Eastern Railway Company; Henry de Mosenthal, F.I.C., Chemist to Nobel's Explosives Company, Limited. As associate: Edward Halliwell, assistant to Mr. Fairley.

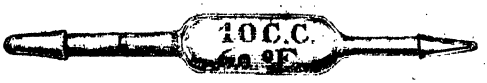
A SPECIFIC GRAVITY PIPETTE.

By W. F. KEATING STOCK, F.I.C.

(Read at the Meeting, January 13, 1897.)

I HAVE lately found the little instrument described below of great use in cases where the gravity of small samples of castor-oil, glycerin, and other matters of high viscosity was required. It consists of a 10 c.c. pipette, with the bulb occupying a central position on the stem. The bulb is of the usual elongated form, but instead of being

cylindrical, it has a flat side, which gives it a stable seat on the pan of a balance. It is six inches in length, and each end of the stem is closed by a ground glass cap, having a good long hold on the stem. The graduation mark is placed one inch below the upper orifice of the stem, and the cap on the lower end is ground so as to fit within 1 m.m. of the point. The point is of such a bore that glycerin passes freely at ordinary temperatures under slight pressure. To use the pipette a piece of suitable size indiarubber tubing is slipped over the open upper end, the liquid to be tested is brought to the required temperature, and the pipette is filled through the pointed end by sucking at the indiarubber tube; when the liquid has risen just above the mark the point is closed by a finger, the indiarubber tube is removed, and the first finger of the right hand is placed over the orifice, the pipette being then held, as usual, between the thumb and second finger. The lower part of the stem is then freed from liquid by wiping with soft filter paper. The liquid is adjusted to the mark and the lower cap is put on. A little roll of filter paper is now used to remove any liquid adhering to the inner walls of the tube above the graduation mark, the upper cap is put in place, and the whole instrument is then wiped clean with a soft cloth, and is ready for weighing. It is not my wish to compare it with any other form of specific gravity apparatus. I have found it easy to use, easy to clean, and accurate. My own instrument was made by cutting down a 10 c.c. pipette and fitting it with caps, etc., in the laboratory. Messrs. Brady and Martin, of Northumberland Road, Newcastle on-Tyne, have asked to be allowed to reproduce it in convenient sizes (probably 5, 10 and 25 c.c.), and they will probably have the pipette for sale shortly, under the name of "Stock's Specific Gravity Pipette."



Discussion.

Mr. Richmond desired to know how Mr. Stock ascertained that the liquid was of the proper temperature within the instrument.

Mr. Stock said he had not determined the difference between glycerin at 15·5° C. in a small beaker and the temperature of the liquid in the pipette when it had taken a few seconds to fill; he did not imagine that the difference would be very great.

NOTE ON THE PRESENCE OF COPPER IN OYSTERS.

By W. F. Lowe, A.R.S.M., F.I.C.

(Read at the Meeting, December 2, 1896.)

A short time ago a few oysters were brought to me by a friend, who said he had reason to believe that they contained copper, as he had examined one or two and found copper in them.

On examination I found this to be the case. In some of the oysters the presence of copper was apparent to the eye, as they were of a bluish-green colour, not all over, but in patches; and in one oyster the whole of the colour seemed to be concentrated in the large muscle used by the animal in opening and closing its shell.

In some of them, however, which also contained a considerable quantity of copper, no bluish colour could be seen, the only difference being that the dark portions of the fish had a somewhat olive-green tint that would not be observed unless they were closely examined.

The shells were free from colour, and no copper was found in them.

The quantity of copper in a single oyster amounted to 40 milligrammes, or about $\frac{6}{10}$ grain, so that but few of them were required to produce very unpleasant results.

They were obtained from the Mumbles, near Swansea, and had probably been laid down in the neighbourhood of some refuse from the copper works.

DISCUSSION.

The PRESIDENT (Dr. Stevenson) remarked that it was easy, during the operation of incineration over a Bunsen flame, to introduce traces of copper from the burner, so that in cases of this kind it was undesirable to use brass burners.

Mr. BODMER said that certain French oysters of a green colour contained less copper than some others whose colour was much less pronounced, so that the colour did not appear to be due to copper at all. It was a well-known fact, however, that oysters did, as a rule, contain copper. In a recent case in which he had been engaged, connected with peas containing copper, it had been brought forward for the defence that a dozen oysters contained rather more than a grain of copper—a quantity somewhat in excess of that contained by 1 lb. of the peas in question.

Mr. HARVEY said that, in his experience, copper was very widely disseminated in shell-fish; but he did not regard the green colour constantly observed in them as having any necessary connection with the presence of copper. He instanced the case of an arrangement of tanks for hatching salmon ova, in which the water was suspected of having dissolved copper from the taps, which, however, was found not to be the fact. However, he had always connected the delicate pink colour of the ova of salmon with the presence of a very minute quantity of copper.

The PRESIDENT said that the oyster was not singular in containing copper. The human body, for instance, nearly always contained small quantities, especially the liver. It might not necessarily be a natural constituent, but was probably derived from certain articles of food, or from cooking utensils.

THE STATEMENT OF ANALYTICAL RESULTS.

BY J. F. LIVERSEEGE, F.I.C.

(*Read at the Meeting, December* 2, 1896.)

THE question I wish to discuss is, not how far should analytical results be given in reports, but to ask what are the principles by which the number of significant figures given in a result should be decided.

The following analytical results have been reported in recent prosecutions under the Sale of Food and Drugs Act:—

I. A whisky was 34·71° under proof.

II. Alcohol, 78·03 per cent. in spirit of nitrous ether.

III. Peas containing 3·11 grains of sulphate of copper per pound.
IV. Butter with 19·88 per cent. of water present.
V. Milk adulterated with 11·77 per cent. of added water.
VI. Coffee adulterated with 68 per cent. of chicory.

The first four are statements of fact, the others are expressions of opinion. If two distillations of alcohol often differ by 0·2 per cent., what is the use of giving a second decimal place? Is there any probability that the analysts of III. and IV. would again obtain 3·11 grains or 19·88 per cent. as the result of other analyses? Ought a milk standard to be used to calculate the added water to 1 per 10,000? If these second decimal places have no meaning, is it wise to appear to claim such a high degree of accuracy? It seems to me to be reasonable to expect that a *second* decimal place should not appear on a certificate, unless it is probable that the *first* could be again obtained in another analysis, and that, unless the unit is certain, adulteration should only be certified to 5 or 10 per cent.: in VI., for instance, report 65 or 70 per cent. of chicory to be present. Suppose that in I. another analyst found 34·91° under proof, some magistrates would consider this a much more serious difference than if one analyst certified 34·7° and another 34·9° under proof. I also think that in V. the analyst would have felt rather foolish if, in answer to questions, he had admitted that there might be 15 or 20 per cent. of water added, instead of the 11·77 per cent. certified to be present.

In water analysis decimals appear very largely; the following are single analyses (not averages) taken from standard works. Total solid matter, 108·88, 220·92, 320·72; nitrogen as nitrates, 6·499, 14·717, 19·858; ammonia, 1·366; organic nitrogen, ·141, ·298, ·531; organic carbon, ·142, 1·792, 2·662; total hardness, 116·9, 140·8.

It appears to me that in each case the last figure of the result is superfluous and should be omitted, the previous figure being increased by one if the last figure is 5 or more. If 250 c.c. of water were taken for the total solid matter recorded as 220·92, the residue would weigh 0·5523 gramme, and each milligramme error would affect the result by 0·4; one decimal place is therefore as much as should be put in any ordinary analysis. In the determination of ammonia, an error of 0·5 in 5 c.c. of ammonium chloride solution is possible, or 10 per cent. I would suggest that to proceed by units from 1 to 25, and then by 5 up to 100, irrespectively of the decimal place, would be better than putting meaningless figures; 1·366 would then become 1·4. When the amount of hardness present exceeds 50, the unit figure becomes very uncertain, and 116·9 may for practical purposes be written 117 or 120. Similarly, nitrogen as nitrates, "6·499," should be written 6·5; and my experience of organic carbon and nitrogen makes me think that the third decimal place cannot be depended on. It is no doubt advisable to record the exact figures found in the laboratory notes, but in reports and printed matter these extra figures appear to me to be unadvisable and misleading.

These superfluous decimals also appear in other chemical work. A recent paper on lemon-juice stated that one fluid ounce contained 34·089 grains of citric acid; the milk analyses giving such specific gravities as "1·03148" are well known, and the following quotations cannot easily be surpassed. "Use a 0·0845691 per cent. solution of K4FeCy6, 3Aq." "The factors for a volumetric solution of iodine are:—Salicylic acid, 0·18132606; thymol, 0·2956772; and β-naphthol, 0·37843106."

In text-books, the meaning of "per cent." is sometimes doubtful; it may be by weight, or by volume, or grammes per 100 c.c. In my own notes, I have for some years been in the habit of using "%" in cases where there can be no ambiguity: of adulteration "%," for instance, and for per cent. *by weight*. Per cent. *by volume* has been expressed by the sign "v/v" (*i.e.*, volume on volume), and grammes per 100 c.c. by "w/v" (*i.e.*, weight on volume). These signs show readily exactly what is meant. Alcohol S.G. 0·838, for instance, 84 % = 88·7 v/v = 70·4 w/v.

Discussion.

The President (Dr. Stevenson) said he thought that, as a rule, insignificant decimal places were better dispensed with, but in some cases it was perhaps a matter of convenience to insert them. For instance, in water analyses, where some of the factors really required the use of three decimal places for their proper expression, it was more convenient to state the results all through to three decimal places than to jump about from two to three. In cases where the third place had no real significance it might be filled by a cipher for the sake of symmetry.

Mr. Allen said that he was in the unusual position of disagreeing with the President, for he thought that, if a fraction were cut short by omitting the concluding figure, the place of this figure ought not to be filled by a cipher, inasmuch as a definite value was thereby assigned to it which was not intended. If the figure was assumed to have no significance whatever, the place ought to be left blank.

There was only one excuse for the use of these long decimals, and that was that the chemist gave the results he got. He put them forward, not as the strictly correct results, but as the actual results of his work, and if these results required a decimal fraction to express them, they should be stated accordingly. That was the only excuse, except in the case of gold and steel analyses. The third decimal place in a steel analysis had a distinct significance in the case of phosphorus and sulphur, and affected the second place, where any variation became of consequence. He was afraid the public sometimes looked upon analysts as quacks, more or less, because they reported to two or three places of decimals in cases in which it was exceedingly doubtful if they would get on repetition a result agreeing even to the first place of decimals. The Society itself occasionally offended in this respect, for, in the circulars recently issued to members respecting the form of milk certificates, and which was made necessary by the decision in the case of Fortune *v.* Hanson, the sum of the results of a milk analysis was directed to be stated to two decimal places! He (Mr. Allen) felt very strongly that it was a mistake to pretend to express results with extreme accuracy unless such accuracy could be approached reasonably closely; and he thought that, except in special cases like those he had mentioned, the sooner the practice was discarded the better.

Mr. Cassal thought there was a great deal to be said on both sides of the question. He agreed with a good deal of what Mr. Allen had said, and to a certain extent with the author of the paper, but it had to be remembered that public analysts were not their own masters in all matters of this sort. Mr. Allen had alluded to milk, and had caused some laughter by mentioning the fact that the statement of results to the second decimal place had been suggested in the Society's circular; but what was one

of the effects of the case of Fortune *v.* Hanson? It was this: If public analysts wished to avoid being put to serious inconvenience, and wished to avoid following the practice which in his student days was known as "cooking," the percentage of added water would have to be stated to a decimal place in most cases. If the percentage of added water was stated to the nearest whole number, as used to be done, it would be necessary to alter the percentage of non-fatty solids, which had now to be given on the certificate, for it necessarily happened that in most cases the percentage of added water calculated from the non-fatty solids ran into a decimal. It was not necessary, of course, to go to the second place of decimals.

With regard to water analyses, he thought that absolutely unnecessary figures ought not to be stated at all, though in one place he had been for a long time practically compelled, through no fault of his own, to state the result of certain water analyses in parts per 100 million (laughter). It was done originally, he believed, because it was thought that those who had to consider the figures understood whole numbers, but did not quite appreciate decimals, and the result was that they were provided with "parts per 100 million" because they had then got something to catch hold of (laughter).

The real way out of the difficulty was to apply common sense to the matter. It was one which required to be dealt with according to the individual merits of every case in which it arose.

Mr. BEVAN said that the case of Fortune *v.* Hanson had been to a large extent set aside by the decision in the case of Bridge *v.* Howard. In that decision it was clearly shown—at least the judge allowed it to be understood—to be quite unnecessary to state the quantity of added water to a decimal fraction. The percentage of solids-not-fat in the case of Bridge *v.* Howard was 7·95, and the added water, calculated to one decimal place, was 6·5. He (Mr. Bevan) had called it 6 per cent., and the judge, having made the calculation for himself, said that as a matter of fact the percentage of added water had been under-stated, but took no exception to this.

Mr. CASSAL said that his experience was that the accuracy of an analysis would be doubted by non-experts if, when the figures were asked for, the actual figures, including the decimals, were not given. He might say that on one occasion a magistrate wanted him to leave out the decimal figures in the non-fatty solids reported in his certificate in order that the method of calculating the amount of added water might be made "more clear."

The PRESIDENT said that Mr. Cassal's experience appeared to be exceptional. He himself had been in the habit for twenty-five years of stating whole numbers only, without fractions, in the case of milk. In regard to certain other things, such as coffee, he had taken still greater latitude, as he would not venture to distinguish between, say, 55 and 60 per cent. in the case of coffee adulterated with chicory, and he had found magistrates sufficiently reasonable to see the force of this.

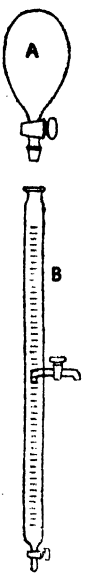
He was glad to think that the difference of opinion between Mr. Allen and himself was, after all, but a small one.

A MODIFIED SCHMID PROCESS.

By R. W. Woosnam.

(*Read at the Meeting, January* 13, 1897.)

The difference of a milligramme in the weight of butter-fat obtained by the usual processes from 5 c.c. of milk is often of much greater importance than would be supposed at first sight. Especially is this the case with separated milks, where a variation of 0·02 per cent. (the result of a difference of one milligramme in the weight) may sometimes even constitute the margin between a good and an only moderate separation. In butter-factories, where large quantities of milk are dealt with, this difference often represents a considerable item in pounds of butter.



In order to ensure a more exact result, without at the same time unduly prolonging the process, I have devised the piece of apparatus as shown in the accompanying figure. It is constructed in two parts, as follows:

(A) A boiling-flask, with tap, the mouth ground to fit the top of B.

(B) A graduated 100 c.c. burette, with small tap at the side, placed preferably at about the 50 c.c. mark.

In making a determination, 25 c.c. of the milk are pipetted into the boiling flask A, and about 28 c.c. (roughly) of strong hydrochloric acid added. For this purpose, a 25 c.c. pipette, with stem sufficiently small to pass through the tap, is recommended. The flask is then placed in boiling water, and agitated frequently until the contents assume a *pale* brown colour. This will generally take place in about two minutes, and it is important that the action should not be allowed to proceed too far, or the sugar caramelized will be, to some extent, extracted by the aqueous ether at the next stage. The flask is now cooled thoroughly, and 50 c.c. of ether added. The tap with which the flask is fitted is then closed, and the whole shaken vigorously for some moments, after which it is fixed firmly into the top of the burette B, the ground-surfaces making a tight joint. When the ether layer has separated, the tap is turned on, to allow the liquids to flow gently into the burette. The measurement of the ether layer is next read off, and, after adjusting the levels of the liquids by manipulating the two taps of the burette, an aliquot part of the ether layer is drawn off from the side-tap into a suitable weighed vessel, the ether driven off, and the fat weighed in the usual manner.

The results obtained have been very satisfactory.

The advantages claimed are: That a larger quantity of milk may be worked upon; that the tapped boiling-flask lends itself more perfectly for thoroughly shaking with the ether; that the reading of the ethereal layer is more exact; that, when desired, more than one aliquot part of the fatty ether may be drawn off; and, lastly, that greater accuracy is obtained than by the old methods.

The apparatus is made by Messrs. Müller, of 148, High Holborn.

THE DISTILLATION OF FORMALDEHYDE FROM AQUEOUS SOLUTIONS.

By Norman Leonard, B.Sc., Harry M. Smith, and H. Droop Richmond.

(*Read at the Meeting, February* 3, 1897.)

In a paper (Analyst, xxii., 5) two of us (L. and S.) have given the results of distillation of aqueous solutions of formaldehyde, expressed as grammes of formaldehyde distilled in given fractions. In the Analyst (xx., 193) one of us (R.) has shown that when dilute solutions of the fatty acids of the acetic series are distilled, the quantity of acid found in the distillate is expressed by the formula

$$100 - y = \frac{(100 - x)^a}{100^{a-1}},$$

provided that condensation in the retort does not take place.

y = percentage of total acid in distillate.

x = volume of distillate expressed as percentage.

It was further shown that a is the rate of distillation—*i.e.*, the ratio of the percentage of acid in the vapour (*i.e.*, parts of acid per 100 of vapour) to the percentage of acid in the solution in the retort (*i.e.*, parts of acid per 100 of solution).

We have examined the results yielded by the distillation of formaldehyde, and find that the quantity of formaldehyde distilled is expressed by the formula

$$100 - y = \frac{(100 - x)^{1\cdot65}}{100^{0\cdot65}}.$$

The annexed table gives the results found compared with those calculated by the formula; it is seen that the agreement is very close:

Vol. of Fraction Distilled.	Formaldehyde in Fraction.							
	Found	Calc.	Found	Calc.	Found	Calc.	Found	Calc.
0 – 20	·0024	·00240	·0023	·00234	·0026	·00237	·199	·208
20 – 40	·0021	·00204	·0018	·00199	·0020	·00201	·180	·177
40 – 60	·0017	·00164	·0017	·00160			·139	·142
60 – 80	·0011	·00117	·0012	·00114	·0031	·00334	·105	·1015
80 – 100	·0005	·00055	·0006	·00053			·053	·0475
Total ...	·0078	·00780	·0076	·00760	·0077	·00770	·676	·676

We may again point out that the volatility of a substance in dilute aqueous solution is entirely independent of the properties of the substance in a pure state, and is solely dependent on its solubility in water. The following table of rates of distillation and properties will show this:

Name.	Rate of Distillation.	Properties in Pure State.
Formaldehyde ...	1·65 ...	Gas, b.-p.,—21°
Formic acid ...	·4 ...	Liquid, ,, 100°
Acetic acid ...	·667 ...	,, ,, 118°
Propionic acid ...	1·111 ...	,, ,, 137°
Butyric acid ...	2·0 ...	,, ,, 162·5°

THE COMPOSITION OF MILK AND MILK PRODUCTS.

By H. Droop Richmond.

(*Read at the Meeting, March* 3, 1897.)

Of the 32,362 samples examined in the laboratory of the Aylesbury Dairy Company during 1896, 26,723 consisted of milk.

The average composition of 11,633 samples taken from the railway churns on arrival at the depôts of the company is given in Table I.

Table I.—Average Composition of Milk Inwards.

Month.	Morning Milk.				Evening Milk.							
	Specific Gravity.	Total Solids.	Fat.	Solids-not-Fat	Specific Gravity.	Total Solids.	Fat.	Solids-not-Fat.	Specific Gravity.	Total Solids.	Fat.	Solids-not-Fat.
January	1·0327	12·76	3·71	9·05	1·0324	13·16	4·10	9·06	1·0325	12·96	3·91	9·05
February	1·0327	12·63	3·61	9·02	1·0324	13·02	4·00	9·02	1·0325	12·82	3·80	9·02
March	1·0327	12·63	3·61	9·02	1·0323	12·96	3·95	9·01	1·0325	12·81	3·79	9·02
April	1·0327	12·58	3·56	9·02	1·0325	12·93	3·90	9·03	1·0326	12·77	3·74	9·03
May ...	1·0328	12·42	3·40	9·02	1·0323	12·76	3·79	8·97	1·0326	12·61	3·61	9·00
June ...	1·0323	12·31	3·42	8·89	1·0318	12·55	3·72	8·83	1·0320	12·43	3 57	8·86
July ...	1·0316	12 24	3·50	8·74	1·0312	12·50	3·80	8·70	1·0314	12·37	3·65	8·72
August	1·0315	12·40	3·65	8·75	1·0313	12·69	3·96	8·73	1·0314	12·55	3·81	8·74
September	1·0321	12·61	3·71	8·90	1·0318	13·07	4·15	8·92	1·0319	12·84	3·93	8·91
October	1·0328	12·83	3·75	9·08	1·0324	13·23	4·17	9·06	1·0326	13·03	3·96	9·07
November	1·0329	12·89	3·78	9·11	1·0325	13·27	4·17	9·10	1·0327	13·08	3 97	9·11
December	1·0327	12·87	3·80	9·07	1·0324	13·24	4·17	9·07	1·0326	13·06	3·99	9·07
Average ...	1·0325	12·60	3·63	8·97	1·0321	12·95	3·99	8·96	1·0323	12·78	3·81	8 97

The fat in all these samples has been estimated by a centrifugal process of the Leffmann-Beam type, various centrifugal machines having been used; the solids-not-fat and total solids have been in the bulk of the analyses calculated by the formula given in a previous report (Analyst, xix. 65, cf. also xx. 57). In 100 test experiments the difference between the results calculated and those found has not exceeded 0·15 per cent.

The following analyses are of abnormal milks:

	I.	II.
Specific gravity	1·0292	1·0303
Total solids	11·57	12·42
Fat	3·51	4·00
Milk-sugar	3·41	3·71
Proteids	3·84	3·89
Ash	0·81	0·82
Solids-not-fat	8·06	8·42

A further sample (not authenticated) was received from the same source as No. I., and was sour when analysed. The results were:

Total solids	11·17	
Fat	3·65	
Proteids	3·36	containing nitrogen ·528
Ash	0·71	
Solids-not-fat	7·52	

I would draw attention to the fact that the best method of analysing sour milks is to estimate fat by Werner-Schmid method, total nitrogen by Kjeldahl method, and ash; these three data, taking 3·0, ·50, and ·70 as limits respectively, are, in my opinion, sufficient to show whether the sample be genuine or adulterated. No reliance can be placed on an estimation of the solids-not-fat in a sour milk, because it is in itself unreliable; differences of over 1 per cent. were found in the total solids of a sour milk, between estimations in one case drying till the loss was less than 1 milligramme in one hour, and in the other till the loss was less than 3 milligrammes in three hours.

It has long been known that milk received on Mondays is poorer than that received on other days of the week. The following table gives the average percentage of fat found on each day of the week; it is deduced from a series of 5,414 analyses, and is chosen, not because it represents the average composition of milk, but because the results are comparable from day to day.

Day.	Percentage of Fat.
Sunday	3·74
Monday	3·70
Tuesday	3·78
Wednesday	3·75
Thursday	3·75
Friday	3·75
Saturday	3·73
Average of week ...	3·74

The average composition of clotted cream was:

	Maximum.	Minimum.	Average.
Total solids	75·18	56·42	67·64
Fat	68·23	46·66	59·16
Ash	0·90	0·52	0·68
Solids-not-fat	11·31	6·40	8·48

The following percentages of water in butter have been found during the year:

Description.	Maximum.	Minimum.	Average.
English fresh butter ...	15·18	12·40	13·82
,, salt ,, ...	17·92	10·46	13·94
French fresh ,, ...	15·94	11·76	14·40
,, salt ,, ...	14·35	9·35	12·21

The samples of English butter were all examined shortly after churning; it is seen that there is practically the same percentage of water in fresh and salt butter. The sample containing 17·92 per cent. of water was a very small quantity, churned for a special purpose, and was not worked to the same extent as usual. Two other samples of salt butter contained just 16 per cent. of water, and the evidence obtained this year goes to show that the limit of 16 per cent. which has been proposed for water in butter is a reasonable one.

A series of determinations of the refractive index of butters prepared in the laboratory was carried out. The following readings were obtained with Zeiss' butyro-refractometer at 35° C.:

Maximum.	Minimum.	Average.
48·5	45·2	46·5

In connection with this, it is interesting to record a few other figures:

	Maximum.	Minimum.
Fat of colostrum of women ...	55·4	52·8
,, human milk	55·4	48·2
Olive-oil	56·8	56·6*
Cotton-seed-oil	61·8	61·4*
Palm-kernel-oil	...	41·2
Margarine	...	52·0

THE DETECTION OF MIXTURES OF DILUTED CONDENSED OR STERILIZED MILK WITH FRESH MILK.

By H. Droop Richmond and L. K. Boseley.

(*Read at the Meeting, March* 3, 1897.)

There are three methods which may be employed for the detection of mixtures of diluted condensed or sterilized milk with fresh milk.

The first of these, which was pointed out by Faber (Analyst, xiv. 141), depends upon the state in which the albumin exists. It is probable that it is in combination with a base, as on heating milk no coagulation of albumin takes place, but on acidifying, or saturating with magnesium sulphate, the albumin separates with the casein. The albumin appears to be changed from a soluble to a colloidal form.

Secondly, as we have before pointed out (Analyst, xviii. 174), if milk has been heated for some length of time, the rotary power of the milk-sugar undergoes a serious reduction, the cupric reducing power not changing to any appreciable extent.

Thirdly, we propose to describe a method that may be of some use in conjunction with the foregoing. It is based upon the fact that the cream rises with extreme slowness—in three hours practically no cream is observed on the surface of sterilized or diluted condensed milk, and in six hours the layer is only about one-tenth of that given by new milk. If sterilized milk be allowed to stand for twenty-four hours, or more, the bulk of the cream will rise to the surface, but the quantity will be less than that yielded by new milk. The cream will, however, contain a distinctly larger percentage of fat, about 40 per cent., as against less than 30 per cent. in the cream yielded by new milk.

The following figures will illustrate this fact:

Sterilized Milk Allowed to Stand for Six Hours.

	No.	Per cent. of Fat in Milk.	Per cent. Cream Risen.	Per cent. Cream for 1 per cent. Fat.	Per cent. Fat in Cream.	Per cent. Fat in Skim Milk.
	1.	4·30	1·3	·30	23·3	4·05
	2.	3·80	0·7	·18	22·3	3·67
	3.	4·25	1·8	·42	20·6	3·95
	4.	4·10	1·9	·46	24·7	3·70
	5.	5·35	2·8	·52	31·4	4·60
Diluted condensed.	6.	3·62	0·3	·08	—	—

* With sodium light.

STERILIZED MILK ALLOWED TO STAND FOR TWENTY-FOUR HOURS.

No.	Per cent. Fat in Milk.	Per cent. Cream.	Per cent. Fat in Cream.	Per cent. Fat in Skim Milk.
1.	4·30	7·0	46·8	1·10
2.	3·80	6·0	41·8	1·37
3.	4·25	8·8	39·0	·90
4.	4·10	8·7	41·0	·58
5.	5·35	11·1	41·4	·85
6.	3·62	0·8	—	3·48

NEW MILK ALLOWED TO STAND FOR SIX HOURS.

No.	Per cent. of Fat in Milk.	Per cent. Cream.	Per cent. Cream for 1 per cent. Fat.	Per cent. Fat in Cream.	Per cent. Fat in Skim Milk.
1.	4·05	9·2	2·27	17·4	2·70
2.	4·20	11·2	2·66	16·5	2·65
3.	3·90	9·8	2·51	15·9	2·60
4.	3·70	9·8	2·69	18·0	2·15
5.	4·45	13·5	3·03	16·8	2·30

The samples numbered from 1 to 5 were yielded by the same cows in each case.

Condensed unsweetened milk, which has been diluted to the original volume with water, has all the analytical characteristics of sterilized milk, and there appears to be at present no method of distinguishing between them.

To distinguish new milk, on the one hand, from milk which has been sterilized on the other, the following methods may be employed :

(i.) Place 100 c.c. of milk in a graduated cylinder, and allow it to stand for six hours at 60° F. (15·5° C.) ; note the percentage of cream. If less than 2·5 per cent. of cream for each 1 per cent. of fat in the milk has risen to the surface, the milk may be considered suspicious ; if the quantity of cream falls markedly below 2 per cent. for each 1 per cent. of fat, it is highly probable that sterilized milk is present.

(ii.) Estimate the albumin by the method of Hoppe-Seyler, or, better, those of Sebelein or Duclaux. If less than ·35 per cent. is found, sterilized milk may be considered to be present.

(iii.) Estimate the milk-sugar by the polariscope, and also gravimetrically in duplicate ; if the difference between the two estimations be more than ·2 per cent., it will be corroborative evidence of the presence of sterilized milk. It is doubtful whether a proportion of sterilized milk much below 30 per cent. could be detected when mixed with new milk.

The proportion of sterilized milk may be deduced from the percentage of soluble albumin by the following formula :

$$\text{Percentage of sterilized milk} = \frac{0{\cdot}4 - \text{Percentage of soluble albumin}}{0{\cdot}4} \times 100.$$

This is based on the supposition that new milk contains 1·4 per cent. of albumin, while in sterilized milk the albumin is removed.

The following is the formula for deducing the percentage of sterilized milk from the cream rising test :

$$\text{Percentage of sterilized milk} = \frac{2{\cdot}5 - \frac{C}{F}}{2{\cdot}2}.$$

The estimation of albumin is the most reliable test. There are many causes which influence the rising of cream, such as the temperature to which the milk has been warmed or cooled, the size of the fat globules, which varies with the stage of lactation, and the acidity of the milk, and quantitative deductions may be occasionally misleading.

It must be remembered, however, that no sharp distinction can be drawn between milk which has been raised to a temperature over 70° C. for a short period, and which naturally is not sterilized in the true sense of the term, and milk which has been heated for a sufficient length of time to destroy all microbial life. For this reason a milk should not be reported as sterilized solely on the result of a very low percentage of albumin if neither the "creamometer" nor the milk-sugar tests give corroborative indications. It is probable that the milk in this case has been merely scalded.

The following figures by C. H. Stewart (*Brit. Med. Journ.*, 1896, 626) show the percentage of albumin found in milk raised to various temperatures:

Time of Heating.	Soluble Albumin in Fresh Milk.	Soluble Albumin in Heated Milk.
10 min. at 60° C.	·423	·418
30 ,, ,,	·435	·427
10 65° C.	·395	·362
30 ,,	·395	·333
10 70° C.	·422	·269
30 ,,	·421	·253
10 75° C.	·380	·07
30 ,,	·38	·05
10 80° C.	·375	none
30 ,,	·375	none

The following analyses will show to what extent the method can be depended upon:

							Percentage Sterilized.		
No.	Fat.	Per cent. Cream.	Cream Fat.	Albumin.	Milk-Sugar.	Milk-Sugar Pol.	Actual.	Cal. Cream.	Cal. Albumin.
1.	3·86	7·2	1·87	·30	4·85	4·65	33	29	25
2.	4·10	9·6	2·34	·34	4·79	4·68		Genuine	
3.	3·90	7·5	1·92	·29	4·79	4·64	28	26	27
4.	3·70	4·3	1·16	·16	4·75	4·57	56	61	60
5.	4·10	7·4	1·81	·26	—	—	30	31	35
6.	4·00	7·5	1·88	·27	—	—	30	28	32
7.	3·75	7·9	2·11	·35	—	—		Genuine.	

The analyses marked 2 to 7 were made upon mixtures the composition of which was unknown to the operator, those marked 2 to 4 being the work of one of us (R.), those marked 5 to 7 of the other (B.). The results arrived at by analysis are given in the columns marked "calculated," and the agreement between the two methods and with the actual composition is extremely satisfactory.

THE ESTIMATION OF MILK-SUGAR IN MILK.

By H. Droop Richmond and L. Kidgell Boseley.

We have been in the habit of estimating the milk-sugar in milk by the method described by Vieth (Analyst, xiii. 63); this is a slight modification of that worked out by Wiley (*Amer. Chem. Jour.*, vi., 289), and the novelty in it consisted of the making of corrections for volume of fat, etc.

Vieth's method of calculation is founded on the following assumptions: (*a*) That the volume of proteids in 100 c.c. is 3·0 c.c.; (*b*) that the volume of fat in 100 c.c. is the percentage of fat divided by ·93.

This last assumption is not strictly correct, the volume of fat being the percentage of fat multiplied by the specific gravity of the milk and divided by ·93, but the error is very small. It is usually sufficient to multiply the percentage of fat by 1·11 to obtain the volume in 100 c.c. of milk.

Wiley and Ewell have recently published (Analyst, xxi., 182) a method for the estimation of milk-sugar in milk by double dilution and polarization; they make up 65·82 grammes of milk to 100 c.c. and 200 c.c. respectively, precipitate with acid mercuric nitrate and polarize the filtrates.

If a equal the reading from the 100 c.c. solution, and b the reading from the 200 c.c. solution, the correct reading will be $\frac{ab}{a-b}$ and the volume of the precipitate $\frac{100\,(a-2b)}{a-b}$.

A series of thirteen experiments to prove the correctness of the method is given, and the volume of the precipitate is calculated in each case; from the series it is concluded that "this correction is less in quantity than the combined volume of the fat and albuminoids." It appears, however, on recalculation of their figures, that the volume of the precipitate is wrongly calculated, the formula $\frac{100\,(a-2b)}{b}$ having been apparently used instead of the correct one given in their paper, and reproduced above.

In the table opposite Wiley and Ewell's figures are reproduced, the volumes of the precipitate being calculated by the correct formula; the volume of the precipitate is also calculated by Vieth's method.

It is seen that, though the average of the two methods agrees well, there is a fairly wide divergence between individual results. A comparison of the polarimetric figures of Nos. 1 and 9 will demonstrate that this divergence is chiefly due to the large experimental error of Wiley and Ewell's method. In these two it happens that the polarization in the 100 c.c flask is the same, while the polarization in the 200 c.c. flask differs only by ·07, which is within the limits of reading; this difference of polarization makes 1·5 c.c. difference in the volume of proteids. It also makes a difference of ·07 per cent. in the percentage of milk-sugar, while, had Vieth's method been used, a difference of ·07° would make a difference of less than ·02 per cent. To obtain the accuracy recorded, Wiley and Ewell found it necessary to use a very

No.	Per cent. Fat.	Polarization in 200 c.c. Flask.	Polarization in 100 c.c. Flask.	Calculated Volume of Precipitate.	Vieth's Method Volume of Precipitate.	Difference.
1.	—	9·37	19·26	5·3 \ 5·4	5·0*	
2.	—	9·59	20·33	10·7 /		
3.	—	9·36	19·20	4·9 \ 5·1	5·0*	
4.	—	9·60	20·25	10·0 /		
5.	2·9	10·15	20·84	5·1	4·0	−1·1
6.	4·8	10·31	21·21	5·4	5·3	−0·1
7.	3·1	9·49	19·41	4·3	4·1	−0·2
8.	4·0	10·01	20·45	4·1	4·7	+0·6
9.	1·4	9·44	19·26	3·9	2·9	−1·0
10.	5·5	11·05	22·68	5·0	5·8	+0·8
11.	4·4	9·57	19·47	3·3	5·0	+1·7
12.	2·0	9·75	19·93	4·2	3·3	−0·9
13.	17·6	8·72	19·13	16·2	14·4	−1·8
		Mean ...	...	... 5·7	5·5	

delicate polariscope with tubes 400 m.m. long; with a less delicate polariscope and shorter tubes, the experimental error would be correspondingly magnified.

We can only conclude that this method is practically useless, on account of the large experimental error, the waste of time, and large amount of sample required.

The only objection to Vieth's method is that it is necessary to make a calculation for each sample, which, when several determinations are made at once, is apt to become tedious. To dispense with the calculation, we propose to add to each 100 c.c. of milk:

(*a*) Three c.c. of acid mercuric nitrate to compensate for the volume of the proteids.

(*b*) (Fat + 1·11) c.c. to compensate for the volume of the fat.

(*c*) One-tenth of the degrees of specific gravity.

(*d*) A sufficient volume to reduce scale readings to percentages of milk-sugar.

With the instrument we use [the Mitscherlich half-shadow polariscope, described by Vieth (ANALYST, xi. 141)], *d* is 10·0 c.c. to 100 c.c of milk; it may be calculated for other instruments by the formula $\left(\frac{55{\cdot}3\,K \times l}{100} - 100\right) \times S$ where K = factor necessary to convert angular degrees into scale readings, l = length of tube used, and S = specific gravity of the milk (which may be taken as 1·032 without appreciable error).

If the fat was 3·7, and the specific gravity was 1·0325, then $3 + 4{\cdot}1 + 3{\cdot}25 + 10 = 20{\cdot}35$ c.c. must be added to 100 c.c. of milk, or 10·17 c.c. to 50 c.c.

We have noticed the fact pointed out by Wiley and Ewell, that the stronger mercuric nitrate solution prescribed by Wiley is liable to discolour the proteids, but we cannot agree with them that it is the xantho-protein reaction; it is more properly described as Millon's reaction. We find it advantageous, however, to use their weaker solution, and add 15 c.c. of this to 100 c.c. of milk, making up the bulk with water.

* These figures are volumes of sand actually added.

A few test analyses are appended to show that accurate results can be obtained by dilution to obtain direct readings.

				Direct	Dilute	Milk Sugar.		Volume of Precipitate.	
Obs.	Sp. Gr.	Fat.	Dilution.	Polarimetric Reading.		Veith.	Wiley and Ewell.*	Veith.	Wiley and Ewell.*
R.	1·0305	4·50	121·0	4·93	4·17	4·15	3·94	8·0	2·9
B.	,,	,,	,,	4·95	4·21	4·16	3·79	8·0	–·9
R.	1·0340	3·60	120·4	5·85	4·93	4·92	5·07	7·0	9·7
B.	,,	,,	,,	5·88	4·89	4·95	5·48	7·0	16·0
R.	1·0300	4·60	121·1	4·82	4·06	4·05	4·21	8·1	11·6
B.	,,	,,	,,	4·80	4·08	4·04	3·73	8·1	·4
	Average	...	...	5·205	4·39	4·38	4·37	7·7	6·6

We have also experimented with the action of various preservatives added to milk in the proportion of 1 gramme per 100 c.c. (except in the case of formalin where 2 c.c. were accidentally added); the samples were allowed to stand a week in an incubator at 25°.

	Polarization.
Original milk	5·63°
1 c.c. Chloroform per 100 c.c.	5·61°
1 gr. Borax per 100 c.c.	5·50°
2 c.c. Formalin per 100 c.c.	5·62° (corrected for dilution).
1 gr. Salicylic acid per 100 c.c.	5·61°
1 gr. Mercuric chloride per 100 c.c. ...	5·59°

With the exception of borax, it is seen that all the preservatives prevented any change.

Discussion.

The President (Dr. Dyer) said that among the many valuable papers in the old volumes of The Analyst a very prominent place must be given to those which emanated from the laboratory of the Aylesbury Dairy Company in the days of their old friend Dr. Vieth, and it was very pleasurable year by year to find Mr. Richmond carrying on this work, and bringing before the Society the results of his very large experience.

One little point which he noticed in the first paper was the high proportion of ash in two of the abnormal samples of milk. His own experience, and probably that of many others, showed that, when milk gave an abnormally low percentage of total solids, the ash was usually high, at once pointing out the abnormality of the samples, so that they were not easily confused with watered samples.

He was much interested in Mr. Richmond's remarks with regard to the nitrogen in decomposed samples, because he had lately heard from another member of the Society (Mr. Smetham, of Liverpool) that that gentleman had been making a somewhat extensive series of experiments on the value of the nitrogen factor in dealing with decomposed milks. He had not seen all Mr. Smetham's figures, but as far as

* We have, by calculating from our own figures, exaggerated the errors of Wiley and Ewell's method; roughly speaking, we have about doubled the experimental error.

the results had gone some time ago, Mr. Smetham had found that the nitrogen remained remarkably constant. It might be converted into different nitrogenous compounds, but it stayed in the milk, and did not seem to be lost in the form of free nitrogen. He thought that Mr. Smetham would have some results shortly to bring before the Society on this subject.

With regard to sterilized milk, he did not know whether it was suggested by Mr. Richmond that the presence of sterilized milk in ordinary milk was a thing to be deprecated, or regarded as an adulteration. Sterilized milk was generally considered to be a valuable article. It would probably be more interesting to the public to be able to detect the presence of unsterilized milk in milk that was supposed to be sterilized, but to this one would take a shorter cut by making a bacteriological examination.

Mr. ALLEN observed that the multitude of figures which had been placed upon the blackboard, and which, after all, only represented a fraction of the results actually obtained, was a sufficient indication of the vast amount of work the authors of these papers had got through; and as a Society, they were very much indebted to Mr. Richmond and Mr. Boseley for laying before them these results, which had now become a very welcome annual institution, the absence of which would be greatly missed. It was interesting to learn that Dr. Vieth's method for the determination of milk-sugar was able to hold its own with more recent modifications of the process. He had a high opinion of Wiley and Ewell's method; but if the use of a specially delicate polarimeter was essential, it militated greatly against the employment of the method in practice.

Dr. EDMUNDS raised the question whether sterilized milk, and, in fact, all sterilized foods, did not lose some of their vitalizing properties, especially for young children.

Dr. RIDEAL said that he was about to make a similar remark to that made by Dr. Edmunds. He usually lived in the country, but for the last two or three months he had resided in town. It was hardly a chemical matter, but in the country they were in the habit of keeping the milk overnight for the cream to rise. On coming up to London, however, the milk was supplied by a dairy company, and it was found that the cream of this London milk would not rise on standing, which seemed to point to the fact that the mixed milk had not the property of rising as pointed out in the paper. He thought that a consumer buying milk should reasonably expect it to throw the normal amount of cream on standing.

He did not think that either sterilized milk or humanized sterilized milk had the same value as unsterilized milk, which was probably due to the fact that the ferments present had lost their activity. He had been trying humanized milk from the same firm for a week or two, with results that were not satisfactory, but which were improved by the addition of a small quantity of unsterilized cow's milk, which probably supplied the missing ferment, if such were necessary, without materially altering the composition.

With regard to sour milks, he thought that the points referred to by Mr. Richmond ought to be made widely known, as they indicated an easy method of

ascertaining with certainty the composition of sour-milk samples. If a Lister-Babcock machine was used for determining the fat, a Kjeldahl determination could possibly be made on a fair average sample taken from the comparatively large quantity in the machine.

Mr. HEHNER said that the remarks of Dr. Edmunds and Dr. Rideal, which raised some doubt as to the food value of sterilized milk, had a serious aspect in view of sanitary considerations. If there were not any very strong evidence that by sterilization some material damage was done to milk, such statements might do unintentional harm. There was no chemical or physiological evidence whatever, as far as he was aware, that milk did contain any ferment necessary to digestion.

Mr. Richmond had omitted to mention the fact that calcium citrate was deposited from milk on boiling. In condensed unsweetened milks, a notable sediment of citrate existed; and this deposition might perhaps be another means of distinguishing boiled from raw milk. The change in the rate at which the cream rose was a very interesting fact. Whatever the explanation might be, it seemed to him that the viscosity of the milk had become altered. He had thought of using the viscosity of the milk as a rapid means of dairy analysis; but Mr. Richmond's results seemed to show that this would not be possible, except, perhaps, in the case of milks which it was certain had not been boiled. There were, other things being equal, two factors influencing the viscosity of milk—namely, the amount and size of the fat globules, and the amount of solids-not-fat. If the specific gravity and the viscosity, as measured by the rapidity of the flow through a narrow orifice, were determined, which could be done in a minute or two, some useful data might probably be obtained.

Mr. C. G. MOOR, with reference to the question of the adulteration of ordinary milk with unsweetened condensed milk, remarked that there was only one brand of unsweetened condensed milk sold in London to any extent, which was of Italian manufacture, and contained much less fat than it should if made from whole milk, and it contained a large addition of borax or boric acid.

Mr. BEVAN remarked that Mr. Richmond had mentioned only three abnormal samples, and desired to know whether they were the only ones he had met with during the year. He was interested in the President's remarks as to the ash, which showed the very great importance of always determining this, and being guided by it in forming an opinion. With regard to the nitrate test, he had been under the impression that Mr. Richmond had some time ago made experiments in this connection, and had found that nitrates might find their way through the medium of food into the milk in a perfectly normal manner.

Mr. RICHMOND said that the detection of diluted condensed milk in ordinary milk had been considered of such importance by the British Dairy Farmers' Association, that some years ago that Association had offered a gold medal for the publication of a method. Within the time during which the offer held good no method was discovered, but Mr. Faber had since brought out an excellent, and perhaps the best, method. He (Mr. Richmond) and his colleague had also studied the subject, and they thought it might still have sufficient importance to be worthy of consideration. Some years ago he (Mr. Richmond) had read a paper on the discrimination between abnormal and adulterated milks, in which he pointed out that a high ash was charac-

teristic of abnormal milks, and it afforded him much gratification that this was confirmed, as it had been, by the President and by Mr. Bevan. He might say that these three abnormal samples were the only ones that had come under his notice during the year. The question raised by Dr. Edmunds of the physiological effect which sterilization had upon milk was one of extreme importance, and one upon which it was hard to get reliable evidence. The opinions held and positive evidence adduced by medical men on this question were often absolutely contradictory; it was not one, however, which fell within the province of analysts. With regard to Dr. Rideal's remarks on the difference between country and London milk in the matter of the rising of the cream, it certainly was a widely-known fact that milk taken warm from the cow, and set immediately, gave more cream than if it were cooled down to a sufficiently low temperature to keep it for some hours, and not only was the quantity of cream larger, but the cream itself was much thicker. There had been one ferment described as present in milk, namely, the galactozymose of Béchamp, but this observer was the only person who had met with it, and his observations had not yet received confirmation. They had purposely omitted any mention of calcium citrate. They had thought of making some remarks on this subject, but on considering the matter, and making experiments in connection with the estimation of citrates, they found that it was not an easy thing to do, and were unable to get any results of real value, so they thought it best not to fill the paper with points that could only be just touched upon. With regard to the viscosity of milk, he did not think that this factor was likely to be of much use for analytical purposes, as it involved calculations of an extremely complicated character. The viscosity of milk was due to, first, the internal friction of the molecules of the aqueous portion, and, second, to the friction between the fat globules and the serum, and very small variations of conditions might and did affect the ratio of the separate effects of friction.

The PRESIDENT said that the Society's thanks were due to Mr. Richmond and Mr. Boseley for these papers. Even if there did not appear to be any immediate practical object in detecting the presence of sterilized milk, the results obtained were none the less interesting and valuable. The Society was a technical, but not altogether a utilitarian, Society, and always valued facts. Besides, even though the practical application of such facts might not be immediately apparent, there was always the possibility that they might ultimately prove useful.

ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS

FOOD AND DRUGS ANALYSIS.

Distinction between Raw and Boiled Milk. I. Carcano. (*Giorn. di Farm. di Trieste*, 1896, i., 275; through *Chem. Zeit. Rep.*, 1897, 46.)—The sample is warmed gently with a few drops of fairly fresh oil of turpentine and an alcoholic solution of guaiacum added. The appearance of the well-known blue colour shows that the milk has not been boiled, and *vice versâ*. E. H. L.

The Detection of Horseflesh in Sausages, etc. H. Bremer. (*Forsch. Ber.*, 1897, iv., 1-8.)—The various methods which serve for the detection of horseflesh are based either on the glycogen reaction or on an examination of the fat (see ANALYST abstracts, xix., 24; xx., 95 and 252; and xxi., 231). The author gives a summary of the work of others in this direction, and then describes his process, which consists in an examination of the intra-muscular fat.

The meat preparation, from which all visible fat has been removed, is minced in a sausage-machine, and heated for about an hour on the water-bath with water. The fat rising to the surface is poured away with the water, and the flesh, after having been washed several times with hot water, is dried at 100° C. for twelve hours, and extracted for several hours with a petroleum spirit of low boiling-point. Part of the intra-muscular fat thus obtained is taken for the determination of the iodine number, refractive index, and Reichert-Meissl number. The remainder is saponified, the excess of alkali neutralized with acetic acid, and the alcohol evaporated on the water-bath. The soap is dissolved in hot water, a hot solution of zinc acetate (1 part to 2 parts of fat) added, and the zinc soap washed with hot water and alcohol, pressed between filter-paper, and extracted with about ten times its volume of water-free ether for fifteen to thirty minutes under a reflux condenser. After cooling, the solution is filtered into a weighed flask, the ether evaporated, and the iodine number of the residue determined. Every precaution must be taken to prevent access of air during filtration and drying.

The subjoined table gives the results obtained by this method:

	Iodine No. of Intra-muscular Fat.	Iodine No. of Liquid Fatty Acids.
I. Horseflesh sausage without bacon	75·8	108·1
II. Horseflesh sausage with about 6 per cent. of bacon	74·0	104·1
III. Horseflesh cervelat sausage with about 22 per cent. of bacon, well smoked ...	53·7	92·4
IV. Horseflesh cervelat sausage with about 25 per cent. of bacon	74·1	102·1
V. Ordinary sausage with some bacon ...	57·6	94·2
VI. Thuringian cervelat sausage with about 65 per cent. of pig's fat	64·3	95·8
VII. Mixture of I. and V. in equal parts ...	66·4	103·1
VIII. Mixture of IV. and VI. in equal parts ...	65·2	99·5

It is stated that whenever horseflesh is present the petroleum spirit extract has a red to reddish-brown colour, and that even the liquid fatty acids have a more or less reddish-yellow shade. On the other hand, bull's flesh gives a similar colour, so that too much reliance must not be placed on this fact, except as a confirmatory test. When, however, this is found to be the case, when at the same time glycogen is detected, and when the iodine number of the intra-muscular fat exceeds 65, and that of the liquid fatty acids is considerably over 95, there can be but little doubt as to the presence of horseflesh. C. A. M.

Detection of Caramel in Wines—Possible Confusion with the Coal-tar Colours. A. J. Da Cruz Magalhaes. (*Comptes rendus*, cxxiii., No. 21; through *Chem. News.*)—The author found that a caramelized Portuguese liqueur wine responded to the general reactions of coal-tar colours: dyeing mordanted wool in presence of potassium sulphate; forming in presence of lead subacetate an orange-yellow liquid, which yielded up its colouring matter to amyl alcohol, a similar reaction occurring with ammonia in excess, and turning orange-yellow when agitated with yellow mercury oxide. Identical results were obtained with another wine colored with pure caramel from ordinary sugar, as well as in the case of solutions of pure caramel.

Caramels from pure dextrose (1) and saccharose (2) were also tested, with the following results:

Treated with lead basic acetate and amyl alcohol: (1) No coloration; (2) deep orange-yellow.

Supersaturated with ammonia and taken up with amyl alcohol: (1) Greenish-yellow; (2) very deep orange-yellow coloration.

Ether: (1) No coloration; (2) orange yellow.

Mordanted wool: (1) Dyed yellow; (2) orange yellow.

No alteration of the original colour of the solutions was produced by Cazeneuve's test. C. S.

Quantitative Examination of Crude Woody Fibre. Lebbin. (*Arch. Hyg.*, 1897, xxviii., 212; through *Chem. Zeit. Rep.*, 1897, 45.)—This process depends on the fact that ammoniacal hydrogen peroxide dissolves starch and vegetable albumins, but leaves pure cellulose practically untouched. 3 to 5 grammes of the meal or bran are very finely powdered and boiled for half an hour in a large beaker with 100 c.c. of water; 50 c.c. of 20 per cent. hydrogen peroxide are next added, and the boiling continued for twenty minutes, while 15 c.c. of 5 per cent. ammonia are gradually dropped in. The whole is boiled for a further twenty minutes, thrown on a tared filter, and the insoluble matter washed with hot water, dried and weighed. From the residue the weight of ash must be deducted, and also 6·25 times the amount of nitrogen it contains, if there be much present. The process gives uniform results, and the solutions filter well. F. H. L.

ORGANIC ANALYSIS.

Test for Formaldehyde. L. Kentmann. (*Pharm. Gen. Anz.*, 1896, viii., 356; through *Chem. Zeit. Rep.*, 1896, 313.)—If the suspected liquid is floated on an equal volume of a solution of 0·1 gramme of morphine hydrochloride in 1 c.c. of strong sulphuric acid, a red violet colour is produced within a few minutes, provided the formalin exceeds one part per 6,000. F. H. L.

Estimation of Furfuraldehyde by Means of Phloroglucinol. 1. **C. Councler** (*Chem. Zeit.*, 1897, xxi., 2); 2. **C. Krauch** (*loc. cit.*, p. 29).—This is primarily a discussion on the suitability or otherwise of Merck's preparation, "Phloroglucin. puriss. pro anal. (m.p., 210° C.)," for the purpose indicated. On the authority of Krüger and Tollens, Krauch has stated in the last edition of his book, "Die Prüfung der Chemischen Reagentien auf Reinheit," that it is sufficiently pure to be so employed; but Councler holds that it varies greatly in composition, and often contains enough diresorcinol to vitiate the results. He therefore considers it necessary always to use a preparation freed from this impurity by Skraup's method.

The remainder of Councler's paper is also largely polemical, and deals with the modifications of his process, which have been suggested by Krüger and Tollens (*Zeits. angew. Chem.*, 1896, 40). He maintains that it is preferable to wash the precipitate till free from chlorine, and then to dry it to constant weight, rather than to wash with 150 c.c. of water, and to dry at 100° for three or four hours only; and that it is better to add the phloroglucinol in the solid form, in order to keep the volume of liquid as small as possible. He also discusses at some length the question of precipitating the whole of the furfural distillate, or only an aliquot portion thereof, and decides that, in the majority of cases, the latter procedure suffices.

F. H. L.

Gravimetric Estimation of Sugar. G. Ambühl. (*Chem. Zeit.*, 1897, xxi., 137.)—The author recommends that the cuprous oxide produced in the Fehling-Allihn process should be dried for one hour at 98·5° C., and weighed as such, instead of being reduced to the metallic state. He presents an elaborate table, showing the results of the method when applied to forty-six samples of wine, honey, and diabetic urine. In the case of wines, the figures are practically identical with those obtained by weighing the metal, being usually a trifle higher (maximum, +0·09; average, +0·034 per cent.). With honey, the excess averages 0·23 per cent. on amounts of sugar varying from 57 to 69 per cent.; and with urine containing 4·25 to 6·16 per cent. of sugar, the mean difference between the two processes is 0·10 per cent., but in this instance the suboxide was manifestly contaminated with organic substances, which suffer decomposition on ignition. F. H. L.

A spontaneously Inverting Cane-sugar Solution. A. Lam. (*Chem. Zeit.*, 1897, xxi., 56.)—The author has recently examined a sample of English crude sugar which was suspected of adulteration. Immediately after solution it gave in Laurent's

polarimeter a value of +91·0, but in twenty-four hours this fell to 80·5, continuing to alter till after ten days its opticity was +12·5, and after thirty days −24·5 (at which point it remained constant). Titrated with Fehling's solution after standing twenty-four hours, the sample appeared to contain 17 per cent. of reducing sugars (calculated as glucose); but on inverting it according to Clerget's method, the figure (−33·8 at 16·5° C.), taken in conjunction with the positive rotation of 91, showed that it actually contained 92 per cent. of true cane-sugar. Further investigation proved that the anomalous behaviour of the material was due to a rod-shaped micro-organism possessing the property of inverting faintly acid and saline solutions of sugar, but without influence on such as are neutral, alkaline, or free from salt. F. H. L.

The Detection and Extraction of Citric and Malic Acids by Means of Quinine and Cinchonine. L. Lindet. (*Bull. Soc. Chim.*, 1896, 1160-1163.)—Citric and malic acids may be easily distinguished by the difference in solubility in methyl alcohol of their quinine and cinchonine compounds, and on this difference the author has based a process for extracting them from vegetable juices.

Cold methyl alcohol (95 per cent.) dissolves only 0·3 per cent. of acid citrate of quinine and 3·3 per cent. of the neutral citrate, while the corresponding malates are soluble to the extent of about 8 per cent. The presence of malic acid interferes with the precipitation of the acid citrate of quinine, and when, in a mixture of the two acids, the quantity of malic acid is 25 per cent. of the quantity of citric acid, not more than 99 per cent. of the theoretical acid citrate of quinine is obtained. When the proportion of malic acid is increased to 50, 100, and 200 per cent., the quantities obtained respectively are 97, 94, and 83 per cent. Under the same conditions the acid oxalate of quinine (solubility 9·2 per cent.) and the neutral oxalate (solubility 8·2 per cent.) remain in solution, but these also increase the solubility of the quinine citrate.

Cinchonine dissolved in methyl alcohol precipitates malic acid where quinine precipitates citric acid. The solubility of the acid malate of cinchonine is 2·5 per cent., but the other salts of cinchonine are so soluble that the precipitation can be considered as characteristic of malic acid. The acid tartrate of cinchonine dissolves to the extent of 26 per cent., whilst the acid citrate, the acid oxalate, and the acid succinate only crystallize when their solution is evaporated to a syrupy consistency. The presence of tartaric, citric, oxalic, or succinic acids considerably increases the solubility of cinchonine malate in methyl alcohol.

In applying these reactions to the extraction of the acids from vegetable juices, the latter must be evaporated in vacuo, and the residue taken up in methyl alcohol (as concentrated as possible). Tartaric acid, if present, should be removed by adding alcohol and ether and precipitating with potash. The excess of potash is eliminated by precipitating all the acids with subacetate of lead, and afterwards liberating them by sulphuretted hydrogen. This is the method to be adopted when the juice contains much sugar or foreign matter. Tannin must also be removed by absorption with animal matter, such as catgut.

The concentrated acids are dissolved in methyl alcohol, and to a definite quantity

of the solution, containing about 2·5 per cent. of acid, quinine as a powder is added little by little, until after shaking for some time a crystalline precipitate is produced. The quantity of quinine added must not exceed 160 to 170 per cent. of the quantity of supposed citric acid, or the more soluble neutral citrate of quinine will be produced. When the proportion of quinine has been established, the main solution is treated with the calculated amount. After standing for twenty-four hours, the liquid is filtered, and the operation repeated on the mother liquor.

When no precipitate is obtained—that is, when citric acid is absent—malic acid is tested for and extracted in the same way, the maximum amount of cinchonine to be added being 140 to 150 per cent. of the supposed quantity of malic acid. Where the two acids are present together, cinchonine is added to the liquid after precipitation with quinine, since an excess of the latter does not interfere with the precipitation of cinchonine malate.

The acids are easily recovered from the alkaloidal salts by adding ammonia to their aqueous solution, filtering from the alkaloids, precipitating the acids with sub-acetate of lead, and decomposing the precipitate with sulphuretted hydrogen; or the acid may be removed by barium hydrate, the precipitate, after drying, being washed with alcohol, and finally decomposed with sulphuric acid.

In this way the author has extracted the citric acid from the juice of lemons, gooseberries, strawberries, raspberries, and tomatoes, and the malic acid from that of cherries and grapes. C. A. M.

The Iodine Number of Cocoa-Butter. D. Holde. (*Zeits. anal. Chem.*, 1897, xxxvi., 163, 164.)—In an abstract of the work of De Negri and Fabris, "Gli Olii," which was published by the author in the *Zeits. anal. Chem.*, xxxiii., 570, the higher limit for the iodine number of cocoa-butter was given as 51. This high value led Strohl to examine specimens of all the different varieties of the fat (ANALYST, xxi., 231), and the highest figure he could obtain was 41·7. Then Filsinger published a note (ANALYST, xxii., 46), in which he suggested that Strohl's higher limit (41·7) might have been due to the presence of free fatty acids, since in his work with Henking in 1889 he had not found a higher value than 37·5. With regard to this opinion, the author points out that Strohl obtained an iodine number of 39·8 to 40·4 with specimens of fresh cocoa-butter, and that, as he himself has found in the case of fluid fats, and as Späth (ANALYST, xxii., 46) showed in the case of lard, an increase of acidity is accompanied by a decrease in the iodine number. These various communications have led the author to refer to the original paper of Hübl, from whom De Negri and Fabris took their figures for cocoa-butter, and he now finds that those chemists wrongly transcribed the value 51, which in Hübl's paper is 34. C. A. M.

Estimation of Rosin in Fats and Soaps. J. Landin. (*Chem. Zeit.*, 1897, xxi., 25.)—In the hands of the present author, Twitchell's process is perfectly satisfactory; but he prefers to use petroleum spirit instead of ether as the solvent for the resin acids, and also to determine them gravimetrically, owing to the uncertainty

about their true molecular weight. As regards Cornette's process (ANALYST, xxi., 260), sodium resinate is not really soluble in saturated sodium chloride. If an alkaline solution of rosin containing 2 per cent. of colophony is treated gradually with this liquid, the first precipitate disappears, until an equal volume of salt solution has been added, but further additions again increase the amount of insoluble matter. The method is, therefore, not reliable. F. H. L.

Valuation of Glue by Viscosimetry. J. Fels. (*Chem. Zeit.*, 1897, xxi., 56 and 70.)—None of the methods hitherto proposed for testing glue can be deemed trustworthy; and even Stelling's (ANALYST, xxi., 239) is inexact. An exception must perhaps be made in favour of Lipowitz's plan of judging the material according to the consistency of the jelly it yields on treatment with a definite amount of water; but, still, this only gives comparative results. The present investigator holds that by determining the viscosity of a glue solution in Engler's apparatus the actual adhesive power of the substance can be gauged, and, moreover, the figures obtained may be expressed in an absolute form. The process has been in use for a year, and has proved quite satisfactory.

The sample is rubbed to powder, and the moisture estimated by drying it at 100° for two hours: 100 grammes are soaked in about 400 c.c. of cold water for twenty-four hours, melted on the water-bath, and the amount of liquid so adjusted that the solution contains 15 per cent. of dry glue. It is then tested in the viscosimeter at 30° C., the time taken for an equal volume (500 c.c.) of water to run out being considered as unity. Treated in this way, five samples gave the figures: 1·36, 1·60, 1·65, 1·91, and 2·21; and it was found that the values expressed the same differences in quality as were deduced from an examination of the corresponding jellies according to Lipowitz's method. F. H. L.

Mineral Constituents of the Water-Melon. G. F. Payne. (*Jour. Amer. Chem. Soc.*, 1896, xviii., 1061-1063.)—Two medium-sized water-melons cut up and mixed together were found to contain just ½ per cent. of pure ash calculated as free from carbonic acid. The composition of the ash was:

	Per cent.
Sulphur trioxide	4·41
Calcium oxide	5·54
Magnesium oxide	6·74
Potassium oxide	61·18
Sodium oxide	4·31
Silicon dioxide	2·15
Phosphorus pentoxide	10·25
Chlorine	4·94
Iron sesquioxide	0·48
	100·00

C. A. M.

INORGANIC ANALYSIS.

The Separation of Bismuth from Lead. A. L. Benkert and E. F. Smith. (*Jour. Amer. Chem. Soc.*, 1896, xviii., 1055, 1056.)—Herzog (*Zeit. anal. Chem.*, xxvii., 650) has described a method of separating bismuth from lead by precipitation of the former as basic acetate, but the authors consider the method too tedious, and not entirely satisfactory in its results. They have substituted a formate solution for that of the acetate with satisfactory results.

Solutions of lead nitrate and bismuth nitrate in nitric acid were prepared containing in 20 c.c. 0·2076 gramme of lead oxide and 0·1800 gramme of bismuth trioxide respectively. Twenty c.c. of each were mixed in a beaker, nearly neutralized with sodium carbonate, and an excess of sodium formate solution (specific gravity 1·084), and a few drops of aqueous formic acid were added. The total dilution of the liquid was 250 c.c. It was boiled for *five* minutes, the precipitate allowed to settle, and the solution filtered while hot. The basic formate, which rapidly separated when not boiled too long, was washed with hot water, dissolved in dilute nitric acid and precipitated with ammonium carbonate. The ignited bismuth trioxide weighed too much, which was found to be due to lead; but on dissolving the impure oxide in nitric acid and *repeating the precipitation* as basic formate, the results obtained in eight determinations varied from 0·1796 gramme to 0·1806 gramme of bismuth trioxide, as compared with the theoretical 0·1800 gramme.

With a solution containing 0·3600 gramme of bismuth oxide and 0·2076 gramme of lead oxide, two results were obtained—0·3595 gramme and 0·3605 gramme of bismuth trioxide. C. A. M.

Detection and Estimation of Minute Traces of Lead in Waters. J. C. Berntrop. (*Chem. Zeit.*, 1896, xx., 1020.)—The sample, without concentration, is treated with a sufficient quantity of sodium phosphate, the whole well shaken and allowed to rest for twenty-four hours. The precipitate is collected on a filter, dissolved in a little nitric acid, the liquid evaporated on the water-bath till most of the acid has been driven off, and then subjected to the action of sulphuretted hydrogen, If this produces a coloration only, the lead may be determined colorimetrically; otherwise the precipitate may be redissolved in nitric acid and the metal estimated in the usual manner. Three test solutions were prepared, containing 0·1, 0·025, and 0·5 parts of metallic lead per million; both of the former gave a very distinct colour. and in the latter the lead was determined with great accuracy. If the original water is very soft, some calcium chloride should be added before the sodium phosphate.

F. H. L.

Estimation of Manganese in Iron and Steel. L. Schneider. (*Chem. Zeit.*, 1897, xxi., 41.)—If a solution of potassium permanganate containing about 20 milligrammes of manganese is mixed with 300 c.c. of 1 : 2 nitric acid, reduced with a ferrous salt, heated to the boil, and then a small spoonful of lead peroxide added, the reoxidation is instantaneous and complete. This may be proved by cooling the whole rapidly, and filtering through asbestos, when the theoretical quantity of permanganic acid will be found in the filtrate on titration with hydrogen peroxide. It is therefore

evident that the amounts of manganese obtained in nitric acid solution during the analysis of steel may be oxidised to the state of permanganic acid, and estimated volumetrically in a few minutes instead of "hours" (*cf.* Rürup, ANALYST, xxi., 218).

F. H. L.

Estimation of Sulphur in Pyrites with Sodium Peroxide. C. Glaser. (*Chem. Zeit.*, 1897, xxi., 40.)—In carrying out this process—especially when the peroxide is old and damp—it often happens that the insoluble portion of the melt is coloured black, owing to the re-formation of ferrous sulphide from some sodium sulphide present. In such a case it is not necessary to reject the analysis, for the oxidation may be completed in the wet way. The basin and its contents should be cooled somewhat, covered over, and more sodium peroxide added by degrees until the residue becomes brownish red, and a persistent froth appears. The ferric oxide should be washed with boiling water by the aid of the pump; and, as it is easily soluble in brominated hydrochloric acid, it can be tested for sulphuric acid, and the barium sulphate, if any, added to the main precipitate.

Von Asboth has stated that in treating low-grade pyrites (30 per cent.) the barium sulphate may be contaminated with silica, and that it is advisable, therefore, to remove this before introducing the barium chloride. This trouble does not occur with American ores containing 40 to 45 per cent. of sulphur; at least, if the liquid is not allowed to stand after precipitation longer than is required for the barium sulphate to subside (fifteen minutes). F. H. L.

Determination of Meta- and Pyro-Phosphoric Acids in Dried Superphosphates. Piron and Nay de Mézence. (*Ann. de Chimie Analyt.*, ii. [1], 4-6.)—Owing to the non-indication by the official (ammonium citrate) method of superphosphate analysis of the meta- and pyro-phosphoric acid formed from a portion of the orthophosphate during the drying of the product, manufacturers are disinclined to adopt this method, since it reduces the market value of the article. Having found that the meta and pyro-modifications can be rapidly reconverted into the ortho- form by the aid of heat in the presence of nitric acid or an alkali, the authors propose to modify the official method as follows: 2·5 grammes of pulverized superphosphate are digested for twelve hours with ammonium citrate; then made up to 250 c.c. and filtered, 100 c.c. of the filtrate being saturated and made decidedly acid with nitric acid, heated to near boiling for an hour, and precipitated in the usual manner when cold. The difference between the amount of phosphoric acid so determined and that arrived at by the ordinary method, gives the percentage of pyro and metaphosphoric acids in the superphosphate. C. S.

APPARATUS.

An Improved Capped Reagent Bottle. (*Chem. Zeit.*, 1897, xxi., 139.)—The stopper of this bottle is made of the usual flat-headed shape, but it is hollowed out on the top, thus forming a receptacle wherein hygroscopic or other materials can be placed in order to preserve the contents from moisture or carbon dioxide. The cap fits on the outside of the neck of the vessel in the ordinary manner. It is claimed

that this device prevents the stopper from becoming fixed, and that it is not necessary to grease it. By filling the receptacle with formalin, the bottle may also be employed as a sterilizing apparatus. F. H. L.

LEGAL.

PROCEEDINGS UNDER THE FOOD AND DRUGS ACT.

THE SALE OF ADULTERATED GLYCERIN.

(Reprinted from the "Pharmaceutical Journal" of February 27, 1897.)

AT the Birmingham Police Court, on the 19th inst.,* Frank Clent, of No 1, Cooksey Road, was summoned for selling glycerin not of the substance and quality demanded. The prosecution was conducted by Mr. Hiley (assistant Town Clerk), on behalf of the Health Department, and Mr. Philip Baker, solicitor, defended.

Mr. Hiley informed the Bench that, so far as he was aware, that was the first prosecution in Birmingham with reference to adulterated glycerin, and there were two summonses issued against two persons, who were wholesale druggists, charging them with selling glycerin largely adulterated with dilute glucose syrup. About the end of January, the inspector under the Food and Drugs Act found a considerable number of penny bottles of glycerin being sold in small shops, and inquiries revealed the fact that the article was in many instances supplied by the defendant. On February 4 a dozen bottles were purchased at defendant's shop, and on the article being analysed it was found to contain a large percentage of syrupy sugar, which, instead of possessing healing properties, had no medicinal value whatever. Each of the bottles was labelled "Pure Glycerin." After the summons was issued, defendant called at the Health Department, and admitted he knew the glycerin was adulterated, and asked that the proceedings might be stopped.

Annie Bennett spoke to purchasing a dozen bottles of glycerin from defendant, for which she paid 7d.

Inspector Jones and Chief-Inspector Parker deposed to conversations they had with defendant. Parker said that defendant told him he knew the glycerin was adulterated. Others were doing it, he said, and he was forced to do so.

Dr. Hill, the city analyst, said that the sample he received from Jones proved, on analysis to contain 45 per cent. of dilute glucose syrup, or syrup of starch as it was called. Glycerin * * * * * * * * was a drug endowed with active medicinal properties. It was used as a medicine itself, and as a vehicle for other medicines. Nearly one half of the sample he analysed consisted of the starch he had mentioned, which was a sugar artificially manufactured by chemical means from common starch. It possessed no medicinal properties, and was not a drug in any way. * * * * * * That described by him in his analysis would not be injurious. It would only have half its proper value.

Mr. Baker: Concerning the value, is it a fact that it would be very difficult to sell a bottle of pure glycerin at a penny?

Dr. Hill: Glycerin is cheap enough, about 8d. a pound. It is not a question of cost with me, it is a question of purity.

Mr. Baker asked the magistrates to deal leniently with the defendant, who, since the proceedings were taken against him, had had the misfortune to have his shop and uninsured stock destroyed by fire.

Mr. Ryland (Chairman of the Bench) said that although that might be the first case brought forward, they could not look upon it as the first case of adulteration. Even people possessed of no chemical knowledge must know they must not supply articles adulterated like the one in question. A penalty of £5 and costs was imposed.

* February 19.

THE ANALYST.

MAY, 1897.

PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS.

The monthly meeting of the Society was held on Wednesday evening, April 7, at the Chemical Society's Rooms, Burlington House, the President (Dr. Bernard Dyer) occupying the chair.

The minutes of the previous meeting were read and confirmed.

The following paper was read and discussed: "The Separation and Identification of the Typhoid and Colon Bacilli," by F. Wallis Stoddart.

A paper by Mr. J. F. Liverseege, entitled "Notes on Alcohol," was taken as read.

The following gentlemen were elected members of the Society: Mr. William Crowder, F.I.C.; Mr. John Heron, F.I.C.; Mr. L. de Koningh, F.I.C.; and Mr. A. H. M. Muter, A.I.C.

Mr. Richard Murray was elected an Associate.

NOTE ON WEIGHING OUT FATS.

By Charles E. Cassal, F.I.C.

(*Read at the Meeting, January* 13, 1897.)

In carrying out the Reichert process, it is obviously essential that exactly 2·5 grammes of butter-fat should be operated upon if comparative results are to be obtained in any two or more analyses of the same sample. I have reason to believe that this is not always done by those who have occasion to make analyses of butter, and I therefore mention the fact, although I do not think that the remark applies to any members of the Society. Weighing out, however, appears to be generally accomplished by difference: a dish containing the fat is weighed, and 2·5 grammes are taken out by a glass rod. Weighing by difference is inconvenient, and may easily be the cause of serious mistakes, especially when a large number of samples are under examination. I have used the thin porcelain boats, shown in the accompanying illustration, for directly weighing fats for a considerable time, and I find them to be very convenient. A number of them are ground to the same weight. The boat in which the fat is to be weighed



out is counterpoised by another, and the boat containing the fat is suspended in the neck of the distilling flask by a piece of glass rod, the fat being washed down by heating the alcohol which has been placed in the flask for the saponification.

DISCUSSION.

Mr. ALLEN asked where the porcelain boats could be obtained.

Mr. CASSAL said that they were made by Messrs. Baird and Tatlock. A special mould had been made for them, but this having been done, they could now be obtained fairly cheap.

Mr. STOCK asked if it was absolutely necessary that the boats should be ground to the same weight, or whether the use of a counterpoise was admissible.

Mr. CASSAL said there was no particular reason for their being all of the same weight, except that it saved trouble, and time also, when a number of things had to be weighed out. Of course a metal counterpoise could be used, but he preferred to have the boats all of the same weight.

THE SEPARATION AND IDENTIFICATION OF THE TYPHOID AND COLON BACILLI.

BY F. WALLIS STODDART.

(*Read at the Meeting, April* 7, 1897.)

THE triple problem which forms the subject of this paper has received an immense amount of attention at the hands of bacteriologists, but, as a whole, may at the present moment be fairly described as unsolved. It is true that the typhoid bacillus may now be identified with almost as much certainty as any other micro-organism when isolated in pure culture; but the essential preliminary of first catching the object of pursuit before submitting it to the process of scientific cookery known as cultivation is still little more than a matter of luck under the conditions usually prevailing.

I propose, first of all, to show that nothing better should be expected of the processes now in use; then to describe a method by which the typhoid bacillus can readily and certainly be separated from the colon bacillus, and from numerous other micro-organisms; and, lastly, to point out that the difficulty of identifying the colon bacillus is vastly greater than is generally supposed.

The carefully compiled *résumé* of processes presented lately to this Society by Messrs. Pearmain and Moor relieves me of the necessity of describing at any length the special methods recommended for this particular line of investigation. I may briefly remind you that, after the gelatin-plate process had been found to fail in consequence of the rapid growth of liquefying organisms, it was discovered that the addition of a small proportion of phenol obviated this difficulty; and from that time to the present phenolated media have played a large part in all attempts to isolate the typhoid bacillus, though it has been found necessary to largely reduce the proportion of phenol first used. Later it was suggested by Parietti that the simultaneous addition of hydrochloric acid effected a more complete suppression of interfering

forms, and for some years this modified process was almost universally adopted. It now seems to have fallen into disuse. Still later appeared the potato-gelatin method of Elsner, by which, perhaps, a more conspicuous difference in the naked-eye appearance of the colonies is created, but which retains the fundamental faults of older processes. Lastly, I should mention the proposal of Dr. Klein to concentrate the bacterial contents of a large quantity of water by filtration. The process in vogue at the present time is, then, as follows: The particulate matter collected by filtration through a Berkefeld bougie is distributed in (*a*) gelatin plates, or (*b*) broth tubes, both media having previously received an addition of ·05 per cent. phenol. Any growth in the tubes after incubation is then distributed upon agar or gelatin plates, and suspicious colonies in either series of plates are subcultured.

It is difficult to believe that a process which represents the combined work of so many investigators should tend to increase, instead of to diminish, the already nearly insuperable obstacles to success: yet a little consideration will show this to be the case. For a due appreciation of this it is necessary to bear in mind that the one great, if not insurmountable, difficulty is the presence in all polluted waters and similar materials of an immense number of micro-organisms not separable from typhoid except by plate culture, and not distinguishable except by one or more subcultures. These organisms, amongst which is *Bacillus coli*, are much more resistant to phenol than is the typhoid bacillus, which occupies an intermediate position in this respect between them and the more sensitive water bacteria. In common no doubt with most bacteriologists, I spent much time some years ago in endeavouring to develop this test, and I found that a very small proportion of phenol checked the growth of the typhoid bacillus, which refused to multiply in media containing more than about ·15 per cent. of either phenol or hydrochloric acid, or ·1 per cent. of each. On the other hand, a variety of organisms found in water grew in media containing up to ·3 per cent. phenol, and broth containing ·2 per cent. decomposed spontaneously if exposed for a few minutes to the air. It was evident that organisms possessed of this power of resistance to antiseptics are widely distributed, and would generally appear in abundance when the test is applied to polluted waters and the like. This conclusion accords with the published experience of others.

But there are other considerations which tend in the same direction. The typhoid bacillus multiplies under the most favourable circumstances much more slowly than the members of the group of which *B. coli* is the type. Grown in gelatin plate at 20° C. the colonies of typhoid are about twenty-four hours later in making their appearance. Cultivated in broth at 37·5° C., *B. coli* and its allies multiply from two to five times as fast as the typhoid bacillus. This difference in the growth rate, accentuated, of course, by the addition of phenol to the medium, operates in two ways: not only is the numerical superiority of the non-pathogenic forms materially strengthened, but in the final stage of plate culture valuable space is preoccupied in which typhoid colonies might otherwise develop. This is especially the case with the gelatin plate, where the most valuable portion—the surface—is largely occupied within twenty-four hours by the expanded colonies of *B. coli*.

Lastly, there is good evidence that some of the organisms commonly present in

polluted water will in broth culture inhibit the growth of typhoid. This is not the case with *B. coli*, the two forms apparently growing normally side by side; but with some species the double inoculation of broth results in the growth of the saprophyte only.

Now, it is easy to see that if it is a difficult and often impossible matter to find a few typhoid bacilli in the presence of a huge majority of saprophytic forms by the direct application of the plate process, there is very little chance of doing so when the natural tendency of the latter to improve their numerical superiority is fostered by the conditions of culture, and when the inhibitory powers possessed at least by some of them are allowed full play. Nor are matters at all improved by the preliminary concentration already referred to; for by this step no increase in the proportion of typhoid bacilli can be brought about. On the contrary, it is not unreasonable to suppose that the somewhat violent treatment may have a more injurious effect upon them than upon the hardier forms; and waters that call for examination in this way are almost certain to contain quite as many organisms as can be dealt with in plate culture without concentration. I quite admit the desirability of working upon a larger sample than the minute fraction of a cubic centimetre, which is the utmost one can often use in a single plate; but this must be done at present by increasing the scale of operations, and not by crowding an unmanageable number of organisms into a few plates.

This numerical difficulty has been fully appreciated by many investigators, and has been most forcibly presented by Messrs. Laws and Andrewes in their recent report.

Consider, for instance, the case of a moderately polluted water containing 50,000 microbes per c.c. Of these, possibly 90 per cent. may be suppressed by the addition of phenol, leaving 5,000 to be dealt with by plating out. There would obviously be no advantage in concentrating such a water, since it is impossible to deal satisfactorily with a plate of ordinary size containing 1,000 colonies. It would then be necessary to subculture every one of these colonies, for the naked-eye appearances are not to be relied upon.

On the other hand, if the water is cultivated in phenol broth, a greater proportionate increase takes place of *B. coli* than of typhoid, even if the latter is not suppressed altogether, and the plating-out process becomes less hopeful than before. Probably the most promising modifications of the current method are as follows: Cultivate in phenol-gelatin plate at 20° C. for twenty-four hours, photograph the plate, incubate again, and subculture any colonies that subsequently appear; or make plain agar plates, incubate at 37·5 per cent., and subculture every colony.

The utter hopelessness of the task has been brought home to me on several occasions on which I have unsuccessfully endeavoured to isolate the typhoid bacillus from waters which were beyond any reasonable doubt actually conveying the disease at the time, and which yielded abundant chemical evidence of sewage pollution.

Sharing the general impression which prevails as far as I can gather at the present time, that the key to the situation would be found in a more perfect separation of the typhoid from the colon bacillus, I addressed myself particularly to this point. It was obviously advisable to discard the use of antiseptics, to avail one's self of the almost equally complete suppression of water bacteria effected by cultivation at blood-heat,

to stimulate the growth of the typhoid bacilli if possible without forcing its companions, to remove the former as rapidly and completely as may be from the latter's influence, and to bring about a physical separation of the species. In fact, I set before myself as a model the *maass* culture, used so successfully in the case of the cholera spirillum.

Under the idea—I am speaking now of four or five years ago—that *B. coli* was a comparatively sluggish form, I thought the typhoid bacillus might be induced to walk away from a mixture of the two, and I naturally tried the well-known potato culture, in which the typhoid bacillus rapidly forms a thin, scarcely visible layer, whilst *B. coli* is almost confined to the point of inoculation. Many difficulties, particularly that of getting potatoes of uniform composition, induced me to look for substitutes; and I did actually effect a separation of the forms by moistening filter-paper, or a porous plate, with broth, inoculating the centre from a mixed culture, incubating, and making subcultures from various points nearer the circumference. There was, however, considerable difficulty in just keeping the paper moist without getting condensed water in local accumulations, and there was the further disadvantage of being unable to see what was going on. In my endeavour to find a transparent medium I came upon exactly what was required. I found that if any of the ordinary solid media were inoculated with typhoid, and incubated at a temperature at which it just maintained its consistence, the growth took the form of an opalescence rapidly spreading throughout the medium, whereas *B. coli* remained confined to the point of inoculation as usual. Of a great variety of media tried, I obtained the best results with an agar-gelatin containing ·5 per cent. agar and 5 per cent. gelatin, incubation taking place at 35° C.; but the only essential appears to be that the necessary relation between the stiffness of the medium and the incubating temperature shall be maintained. For this purpose it is necessary to be a little more than usually critical in its preparation. The following method gives satisfactory results:

Place 500 c.c. meat infusion in a beaker of about 1,500 c.c. capacity and weigh; heat over a naked flame until just boiling, then throw in 5 grammes agar cut up small, but not previously wetted, cover with a clock glass, and boil gently, placing the flame a little to one side of the bottom of the beaker. The agar will boil down little by little, and will be kept in rapid rotation so that it cannot stick and burn. Continue until the whole of the agar is disintegrated, which will occupy about two hours, occasionally adding boiling distilled water to replace loss. There will be no discoloration. When the operation is complete, make up to the original weight with boiling distilled water. Add 5 grammes peptone and 2·5 grammes salt, and render distinctly alkaline. Boil up again, place in boiling sterilizer, and allow to cool in it overnight. When cold, turn out the mass, cut off the deposit at bottom, remelt, and mix with an equal weight of 10 per cent. nutrient gelatin.

The agar-gelatin should be poured into dishes or flat-bottomed flasks to a depth of about 5 mm. and allowed to cool slowly, preferably in the sterilizer. In use the centre of the medium is touched with a platinum loop charged with the material to be examined, the flask or dish is enclosed in a much larger one in order to prevent condensation, and the whole is placed in an incubator kept at 35° C. for twenty-four hours. Pure cultures of typhoid so treated produce an opalescence occupying about

two-thirds of the medium; *B. coli* gives a flat plate somewhat thicker and moister than the usual form. If the inoculation is made with both organisms, either from separate cultures or from a mixed culture, we get a flat plate of *B. coli* in the centre, with an opalescent halo of pure typhoid. Plates of this medium, inoculated direct from typhoid stools, gave without difficulty the same pure culture of typhoid bacilli; and I anticipate this will become a valuable diagnostic test as easy of application, though not quite as rapid, as the serum test. It is best applied by putting two or three loops of stool into a little sterile broth, shaking and inoculating as described. Tap-water, also inoculated with a trace of a broth culture of typhoid, or typhoid and coli mixed, readily yielded pure cultures of ty hoid. I may point out that here is a legitimate application of the preliminary filtration; the concentrated particulate matter is just what is wanted for this test. I have abandoned this step, however, in favour of a separation of micro-organisms, and, indeed, all suspended matters by centrifugal action. I find that by the aid of a slight chemical precipitation a moderately pure water can be completely sterilized, and the micro-organisms collected in a small amount of sediment by whirling in a Leffmann-Beam machine. This apparatus, however, does not deal effectively with sewage or badly-polluted waters, and I am now engaged in perfecting this mode of concentration.

I was then in possession of a reliable means of effecting the much-desired separation, but thought it necessary to confirm on cultures from other sources, especially as it was becoming known that there were many varieties of *B. coli*, and it was even maintained that the typhoid bacillus was only one of these varieties endowed with exceptional virulence. I obtained, therefore, several specimens of typhoid and three of *B. coli* from bacteriological friends, besides preparing fresh typhoid from spleens and stools.

The typhoids were all practically identical, answered to all the classical tests, and I may say in two words that I have no doubt the typhoid bacillus is a distinct and definite species, not to be confused with anything I have ever found in water, sewage, or healthy fæces. It was quite otherwise with *B. coli*. The three new pure cultures consisted quite clearly of three distinct species, and amongst them was one that comported itself in agar-gelatin like typhoid. At the same time I was accumulating coli-like forms from waters and sewage, several of which behaved in the same way. In doubt as to the value of my test, I applied to Dr. Escherich, who, as the discoverer of *B. coli*, would be in the best position to settle its true characteristics, and he most kindly sent me pure cultures. The *B. coli* so obtained was not identical with any of the others, but resembled most nearly my original culture (obtained from fæces), and it behaved exactly as described in the agar-gelatin medium. I was therefore confirmed in the value of the test as regards typhoid on the one hand, and *B. coli* with a number of its near relations on the other.

I arrived at this stage rather more than two years ago, and my attention was then naturally directed to the study of those forms which behaved generally like *B. coli*, but which grew in agar-gelatin like typhoid. Unfortunately, as far as my experience goes, there is a similar antagonism between them and typhoid—at least in some cases—to that observed in broth cultures. Certain of these organisms occupy the medium to the exclusion of typhoid. If the opalescent growth consisted of both

organisms in equal or nearly equal proportions, of course the separation could easily be completed by plating out. But this does not appear to be the case, and my work here only revealed a greater depth of complication as regards the isolation of the typhoid bacillus. Still, I think it will be conceded that the satisfactory separation of the typhoid and coli bacilli is of sufficient interest to be worth recording, and was a necessary step in the demonstration of the presence of other forms which may mask the typhoid bacillus if present in water. But incidentally some considerations which appear to me to be of the gravest import in connection with the question of water-supply have forced themselves on my attention.

I have during the past three or four years collected a great number of coli-like organisms from natural waters and similar sources. Now, until lately it was the unscientific fashion to refer to bacilli which answered to some only of the usual tests for *B. coli* as "varieties." Thus, one variety did not coagulate milk, another did not form indol, and so on. Latterly, at the instance, I think, of Dr. Klein, all these have been quite rightly dissociated from *B. coli;* they are evidently distinct species, and so numerous that I have long since ceased to collect them for lack of space. But I have to carry the process of differentiation a step further. *B. coli* in the narrower sense is defined as a short oval or subcylindrical bacillus which grows rapidly on the surface of nutrient gelatin, forming a thin plate with irregular margin; it produces gas in glucose media, coagulates milk in two or three days at 37·5° C., and produces indol in broth culture. There are also some minor and less definite characters, such as paucity of flagella and feeble motility. Now, amongst the organisms isolated in my experiments, I have already found more than a dozen practically indistinguishable by means of the tests alluded to, but which I shall have little difficulty in convincing you are entirely distinct species. There are several features, such as morphological differences under rigidly constant conditions of culture, which I believe are truly specific, but which may not be so regarded by all observers. I shall therefore ignore them for the present, and confine myself to characteristics as to which there can be no such difference of opinion. The appended table (see next page) contains such an account of thirteen species, grouped so as to bring out the chief points of difference. There are one or two of the tests which require a word of explanation.

Touch-culture on Gelatin.—It is well recognised that in gelatin cultures of this class of bacillus the surface colonies only take at all a characteristic appearance; and this is modified by the amount of inoculating material, the length of time that elapses before the growing colony reaches the surface, the propinquity of neighbouring colonies, the temperature of incubation, and the age of the medium. In order to control these influences, I have adopted the following procedure, which I call a touch culture. A growth is lightly touched with the extreme point of a plain needle, which is then applied vertically and as lightly as possible to the surface of a freshly-poured 10 per cent. gelatin plate, and this is repeated until the charge is exhausted. The plates are then enclosed in a larger chamber, and incubated at 20° C. for four days. Very characteristic appearances are thus obtained, which are perfectly constant for the same organism. Typhoid is readily distinguished from all the coli-like forms by the slow rate of growth and the pattern of the expansion. Similarly, the forms exhibited may be divided into three classes according to their behaviour, and some

Designation.	Source.	Touch Culture, 4 days at 20° C.	Halo Test.	Serum Test.	Flagella (maximum number).	Distinctive Characters.
C_1	Fæces	Rather thick irregular expansions, with dentate margins. Diameter on fourth day not exceeding 10 mm.	–	+	5; short, and slightly wavy.	Very limited expansion in touch culture. Halo and serum tests in combination.
C_7	Water		+	+	8; long, wavy.	Most like C_8, but distinguished by absence of marked odour, character of halo, and by micro-examination of agar colonies (well-marked bacilli with threads).
C_{13}	Fæces		+	–	5; long, stout.	Most like C_8, C_9, and C_{10}, but distinguished by limited touch culture and character of flagella.
C_2	Pure culture	Thin expansions, with veined and pitted surface and indented margins. Diameter about 20 mm.	–	–	4; very regular wiry spirals of 6 to 8 turns.	Flagella quite characteristic; broth cultures have great tendency to clear rapidly.
C_4	Pure culture		–	–	1; slightly wavy.	All cultures extremely viscid. Flagella distinctive.
C_8	Water		+	+	5; long, wavy.	Halo culture characteristically thick, with abrupt margin. All cultures very repulsive urino-fæcal odour.
C_3	Pure culture	Very thin expansions, with variable surface and foliated margin. Diameter about 30 mm. Growth similar, though smaller, at 12° C., except C_6 and C_{10}, which do not expand, but form moist white elevations.	+	+ +	3; short.	Serum test. Flagella.
C_5	Pure culture (Escherich)		–	–	0	Distinguished from C_{12} by micro-examination of margin of impression preparation.
C_6	Water		+	–	1; stout, slightly wavy.	Touch culture at 12° C. Flagella.
C_9	Fæces		+	–	5; very wavy.	Agar growths at 20° C. So short as to look like cocci.
C_{10}	Water		+	–	8; long and wavy.	Touch culture at 12° C. Flagella.
C_{11}	Water		+	+	3; delicate.	Touch culture; quite characteristic "matt" surface; much more uniform than others, both in surface and margin, which is almost devoid of indentations.
C_{12}	Sewage		–	–	0	Most like C_5, but constant morphological differences.

individuals, notably C_1 and C_{11}, may be at once identified in this manner. Further, the colonies so formed make most excellent impression preparations, the marginal organisms in which in many cases present constant and distinctive morphological features.

Halo Culture.—This refers to the agar-gelatin culture already described. It divides the organisms into two well-defined groups, and furnishes a definite test for C_8.

Serum Test.—It is, I think, a new observation that several organisms react quite as powerfully with anti-typhoid serum as does the typhoid bacillus itself. As a test for typhoid bacilli, therefore, this process fails, though it does not of course follow that the converse "serum diagnosis" of typhoid is misleading. The mode of application is simple. To 5 c.c. of a twenty-four hours broth culture is added the minimum dose of serum found to give a distinct reaction with a typhoid culture; this is generally about ·25 c.c. C_3 gives a more immediate reaction than typhoid; C_1, C_7, C_8, and C_{11} behave very like the latter.

Flagella.—The number and character of these interesting appendages, commonly, though almost certainly erroneously, regarded as locomotive organs, furnish a useful means of distinction between typhoid and *B. coli*, and to a less degree between the members of the coli group. As long as the staining of flagella was a matter of considerable uncertainty this was not the case; but now that Van Ermengem's silver process makes it possible to get uniformly successful preparations, there is no difficulty in grouping these forms on this basis. Thus, one class will consist of those which exhibit no flagella (C_5 and C_{12}); these are more easily recognised by the complete absence of detached flagella, so conspicuous a feature in typhoid preparations. The second group will include those forms which possess one terminal, rather massive flagellum, bacilli on the point of dividing being sometimes provided with one at each end (C_4 and C_6). The third group embraces those furnished with a fringe of three to eight flagella. One form (C_2) is absolutely distinguished from all the rest by the character of its flagella. It is worth noting that it is much more difficult to distribute the coli-like forms through water than typhoid bacilli, and it is very necessary to be on one's guard against mistaking a mass of pauci-flagellate forms for one multi-flagellate organism. In carrying out this process, and, indeed, in making all cover-glass preparations, it is very advantageous to substitute fixing with formalin for heating; both fixation and subsequent staining are much improved.

Indol and Gas Formation.—These generic tests proved very uncertain in my hands, until I observed that it was absolutely necessary that the broth used should be made from very fresh meat, and that the latter should not be allowed to macerate in water before steaming. With media prepared in this way, there is no need of the addition of glucose in observing gas formation, and the indol reaction is very strong. A very pretty and reliable mode of applying the latter test is to use an agar culture, such as is prepared to demonstrate flagella. If a warm solution of nitrous acid, made by adding 1 part of pure sulphuric acid to 10 parts of 0·02 per cent. solution of potassium nitrite, be poured over such a culture, a pink coloration rapidly appears in the superficial layers.

Now, an examination of this table will, I think, make it quite clear that the

organisms referred to are fairly entitled to be considered distinct species. It should be remembered that most of them have been carried through dozens of generations, and still present the same distinctive features. Yet they all conform so exactly to the published description of *B. coli*, that probably no bacteriologist would hesitate to recognise them as typical colon bacilli; indeed, as already stated, several of them were presented to me as such. Nevertheless, the odds against the correctness of the diagnosis, even with our present limited experience, would be twelve to one.

In fact, the number of known species appears to be determined by the range of our discriminating tests, rather than by their actual limitation in Nature. These tests, too, have been collected and arranged with a view to the distinction between the typhoid bacillus and the group of which *B. coli* is the type, and were not intended primarily as a means of recognising this latter. The positive identification of *B. coli* is one of the most difficult tasks of bacteriology.

But this is more than a bit of systematic biology. We are accustomed to draw most important inferences from the discovery of *B. coli*. Take first the number of pathological conditions with which this organism is said to be associated. We are told repeatedly that it has been found in this, that, and the other morbid condition—in peritonitis especially. I read the other day that, after a cadaver had been roughly placed on the post-mortem table, a discharge took place from the external auditory meatus which proved to be a pure culture of *B. coli*. Apart from the singularity of finding a pure culture of anything under such conditions, there was nothing to show that the extremely delicate operation of identifying *B. coli* had been successfully carried out. And this is a fair sample of dozens of similar records.

But a more important matter to us as analysts is the significance of the presence of *B. coli* in drinking-water. It is pretty generally accepted that the normal habitat of *B. coli* is the large intestine, and that its presence in water in any quantity is a certain index of fæcal pollution. There are two assumptions involved in this view which will repay consideration. The first is that *B. coli* is a definite and readily-recognised species, so that there may be no doubt about its identity when met with. I think I have shown that *B. coli*, as generally understood, undoubtedly comprises at least a great many distinct species, that the positive recognition of the true *B. coli* is extremely difficult, not to say impossible, as yet, and that any record of its occurrence, to be placed beyond question, must be accompanied by much more convincing proofs of its identity than have been adduced in any instance hitherto published.

A second assumption is that *B. coli* does not occur in abundance in organic matters other than animal excreta. The mode in which this very dubious theory has developed is instructive. The bacillus was originally found by Escherich in the excrement of infants fed exclusively on mother's milk. Subsequently other investigators found an apparently identical organism in the intestine of man and other animals—a habitat that was soon extended to air, water, soil, and pretty much everything on the terrestrial globe. No proof of this identity beyond the simple characters given originally by Escherich was advanced, and the almost universal presence of the bacillus has been explained by the dissemination of excrement by natural agencies. It will be seen at once on what a slender thread the connection

between *B. coli* and animal excreta rests, and how untrustworthy is the inference so commonly drawn from its supposed discovery in natural waters. It has never been shown that any well-defined species is so invariably associated with excremental matter that its presence can, with any show of reason, be held to constitute a certain index of fæcal pollution. The true *B. coli*, so far as my experience goes, is not generally, if at all, to be found in the intestine, still less in sewage-polluted waters. And, indeed, the connection between *B. coli* and fæcal matter is not even now accepted in the complete and unreserved fashion which should be the case where such important issues are at stake. Let me refer you to one of our best practical handbooks—the manual written by Drs. Kanthack and Drysdale. If you turn to the directions given for obtaining *B. coli*, you will find the recommendation to add to sterile broth some minced fresh meat, and to incubate for twenty-four hours, when a mixture of organisms, including *B. coli*, will result. There is no mention of sewage, fæces, or similar material.

No; when the history of *B. coli* comes to be written, it will be found to be altogether parallel to that of *B. termo*. In both cases a number of saprophytic organisms possessing a superficial resemblance have been confused together; our processes have for some time been capable of distinguishing between some of the forms previously collected together under the name *B. termo*, which undoubtedly included the several species now massed together as *B. coli;* we are hardly yet in a position to deal equally definitely with the latter, and until we are able to single out a form specifically associated with fæcal matter, and at least as easily recognisable as the tubercle or diphtheria bacillus, we shall do well to avoid such expressions as contain index of fæcal pollution.

It may, of course, be argued with some force that the precise identification of species is a matter of small importance, and that the presence of organisms whose function is to break up organic filth is objectionable in a drinking-water, in so far as it is evidence of the existence of that filth. With this one is not disposed to quarrel, but it is no answer to my criticism, which is directed against the inference that this filth is derived from the intestine of an animal because the associated organisms necessarily originated in this locality. When an assertion of this kind is made, it is time to ask for some evidence in support. The sanitary significance of the minute quantity of pabulum necessary for the vigorous life of these organisms will vary enormously, according to its supposed origin; and at present there is no satisfactory evidence that any of the organisms grouped under the general term *B. coli* afford by their presence specific indication of contamination with fæcal matter.

But lately a further refinement has been made in connection with river-water supplies to large towns, especially the Metropolis. It is said that not only is *B. coli* found in unfiltered river-water, but that it passes the sand filters, as is proved by the discovery of the same organism in the filtered water. I would ask again, How has the identity of *B. coli* been determined? is there any reasonable proof that some of these organisms, which may certainly be found in water completely innocent of admixture with sewage, have not been mistaken for *B. coli?* how is it possible to establish the identity of the organisms in the filtered and unfiltered water, if the only available tests are insufficient to discriminate between not two only, but a dozen or more distinct species.

I have merely touched upon these matters, wishing to hear by way of discussion what are the views of those present; but I would remind you that the tendency to hastily assert the identity of similar organisms has over and over again checked the progress of bacteriology, though in no former instance, perhaps, has the confusion carried with it such important consequences.

Bacilli which grow in broth at 37·5° C., rendering it turbid, and producing indol in twenty-four to forty-eight hours, grow vigorously in phenolated media, coagulate milk in two to three days, produce gas in media containing sugar, do not liquefy gelatin, but develop on the surface into a thin, flat expansion, with irregular margin, and are indistinguishable by ordinary plate, stroke, or stab culture.

Explanation of the accompanying Plate.

Fig. 1.—Halo cultures of typhoid and *B. coli*, half natural size.

a. *B. coli*, pure culture.

b. Typhoid and *B. coli*, mixed culture.

c. Typhoid, pure culture. Condensed water has confused one side of growth.

Figs. 2-4.—Types of Flagella attached to organisms resembling *B. coli*. Van Ermengem's stain, × 1,000.

Fig. 2.—C_6, ditto.

Fig. 3.—C_2, ditto.

Fig. 4.—C_8, ditto.

Discussion.

The President said that, in addition to members of the Society of Public Analysts who had worked on this subject, the meeting was fortunate in having among its visitors several professional bacteriologists, among whom was Dr. Pakes, whom the Society had met before upon a similar occasion, and he would call upon Dr. Pakes to be kind enough to contribute something to the discussion.

Dr. Pakes said he would like to begin by saying with what a large amount of interest he had listened to this paper. He really felt envious of the energy which Mr. Stoddart must have possessed to be able to get through the quantity of work necessary in such an investigation. It seemed rather puerile to commence carping upon details, when such an important matter was at stake, but there were one or two points on which he would like to ask questions of Mr. Stoddart.

In the first place, Mr. Stoddart had said that if typhoid and colon bacilli were planted in broth, a mixed culture was obtained, from which it was possible to separate the typhoid bacilli. His own experience was that, if coli and typhoid were planted together, the coli was obtained at the expense of the typhoid. He had made a large number of experiments in this connection, and had come to the conclusion that, at whatever temperature the organisms were planted, absolutely pure cultures of coli were obtained, it being impossible to recover the typhoid. He had grown at all temperatures, and examined after all intervals of time, and had never been able to recover the typhoid bacillus from a mixed culture of colon and typhoid, when the typhoid bacilli were inoculated in an equal or less quantity than the colon bacilli. It could, however, be recovered when it was in the proportion of, say, three to one.

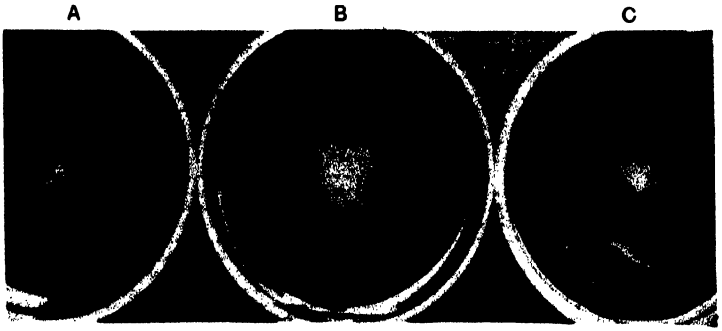


Fig. 1.

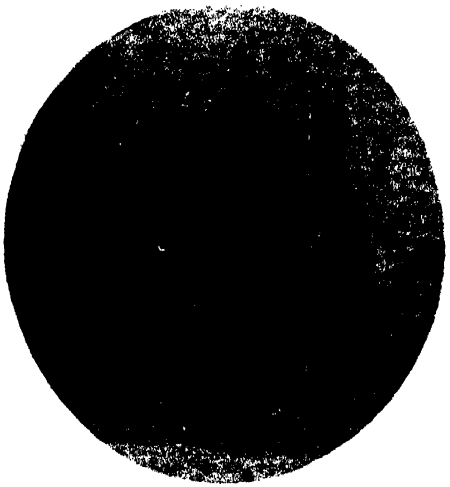


Fig. 2.

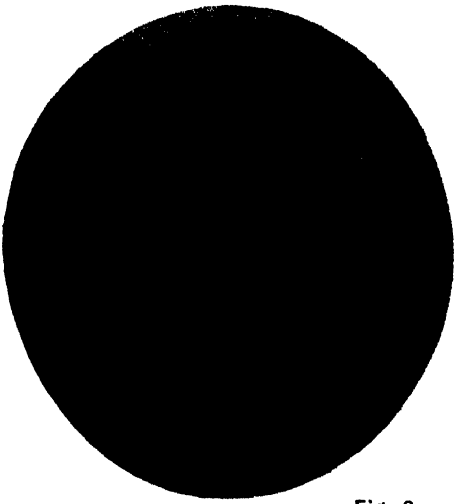


Fig. 3.

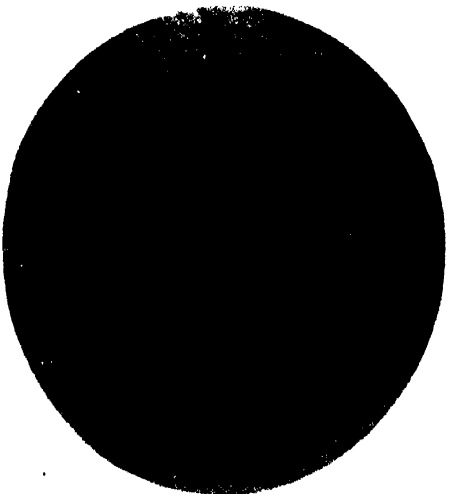


Fig. 4.

Mr. Stoddart had referred to a number of cultures, which he said conformed to the dicta laid down by Escherich as to what the colon bacillus was, but which he (Mr. Stoddart) believed to be distinct from true coli. The speaker desired to know what Mr. Stoddart himself would lay down as the reactions of the true colon bacillus, since all his cultures gave the reactions which Escherich described as pathognomonic of the colon bacillus.

There was one very important factor which Mr. Stoddart had absolutely omitted to mention, viz., the amount of acid produced. It was certainly a reaction which had only been systematically made use of during the last two or three years, but, as Mr. Stoddart would know, the acidity produced by the typhoid bacillus was extremely small in comparison with that produced by most of the organisms that were looked upon as varieties of the colon bacillus.

He (Dr. Pakes) had eleven different cultivations of the colon bacillus, which he had obtained from different sources, actually growing and living. These also conformed to the description of the true bacillus coli; that was to say, they produced the characteristic reactions in broth, with the formation of indol in milk, on gelatine, on agar, and on gelatin agar.

He did not know whence he got the idea of using the last-mentioned medium, but he had employed it for some time. He noticed that Mr. Stoddart had said that 5 + ·5 per cent. gelatin agar would stand a temperature of 35° C. He (Dr. Pakes) had never worked at 35°, but always at 37° C., and he had found that the 5 per cent. medium would not remain sufficiently stiff to enable it to be used at 37°. He could fully bear out what Mr. Stoddart had said with regard to the appearances of the colonies of the two organisms on this medium. Those of the colon bacillus were very much more heaped up, and spread very much less in the same time, than those of typhoid.

With regard to the serum test, as was well known, this test for the diagnosis of typhoid fever was very much to the fore, and during the last three months he had had several opportunities of applying it. It seemed to him that there were some fallacies in the test as applied by Mr. Stoddart. Given the serum of a patient undoubtedly suffering from typhoid, and given two cultures of the typhoid bacillus (which was far more easy to identify than the colon bacillus), one extremely virulent and the other practically non-virulent, it would be found that the former was acted upon better than the latter. Why this should be he could not exactly make out, but it would seem that there was a difference in the action of the bacillus according as it was virulent or not.

He had not heard a word on the subject of the pathogenicity of these different varieties of coli. He had obtained upon different occasions organisms which he certainly thought to be coli, but which, when inoculated into susceptible animals like guinea-pigs, all proved to be non-pathogenic. It would seem, therefore, either that a virulent form of coli existed, or that the organisms in question were not what they were taken to be.

With regard to the different appearances presented by the organisms referred to by Mr. Stoddart, there seemed to be no doubt that certain organisms did vary very largely, in proportion to their virulence, and according to their surroundings. For

instance, the anthrax bacillus, when grown under certain conditions, lost its power of sporulating. Such phenomena were simply dependent upon environment, and it seemed to him possible, if he might suppose for a moment that these organisms did belong to the same species, that such small differences as existed between some of them might have been developed simply by different environment.

These were the points which had struck him in connection with the paper. No doubt Mr. Stoddart had a good explanation to offer for the differences of opinion which he (Dr. Pakes) had given.

He felt that he must express his indebtedness to Mr. Stoddart for a large amount of knowledge gained from the paper, and he desired to put forward the small points on which he differed from Mr. Stoddart, not in any spirit of carping, but with all due deference.

Mr. JOSEPH LUNT, B.Sc., asked whether Mr. Stoddart had succeeded in actually obtaining the typhoid bacillus from naturally polluted water suspected of having given rise to the disease.

He thought Mr. Stoddart had demonstrated that his method, which was a very ingenious and beautiful one, afforded a ready means of separating the typhoid and colon bacilli from artificially mixed pure cultures, and also of obtaining the typhoid organism from typhoid stools, which was a great step in advance; but unfortunately, as Mr. Stoddart showed, there were other organisms which resembled either the typhoid bacillus or *Bacillus coli*, which behaved in the same way as typhoid by his method. It was unfortunate that the typhoid organism could not be obtained absolutely alone. If this could be done, an invaluable advance would have been made. He did not quite understand how Mr. Stoddart distinguished between many of the organisms of the coli group shown in his table. The only differences between some of them seemed to be in the number of their flagella, and it was his experience that just such variations were often obtained in the various individual bacilli in a pure cultivation, so that he thought it would be very unwise to draw any positive deduction from this characteristic alone.

He had been in the habit himself of using such touch cultures as Mr. Stoddart had referred to, and had found that the diameter of the colonies in these cultures was very variable. It might very well happen that one touch would give rise to a colony 20 millimetres in diameter, while another from the same variety would grow only 10 millimetres, owing to the variable amount of sowing material used, and also owing to variations in their vitality. He could not, therefore, agree with Mr. Stoddart that the differences shown by these cultures were very essential ones, unless other characteristics confirmed the indications given by the diameter of the colonies at a certain age.

He therefore did not consider that Mr. Stoddart had fully demonstrated specific differences between all the coli organisms he had regarded as distinct from one another.

He quite agreed that it was rather a hopeless task to find typhoid bacilli when mixed with *B. coli* in large quantity by Parietti's method, in which carbolized media were used, and hoped Mr. Stoddart's method would receive the attention it well deserved.

Mr. C. G. Moor said that he had always been impressed with the necessity for not working upon a tenth or hundredth part of a cubic centimetre, and as the organisms might be injured by the method of concentration (filtering the water through a Pasteur or Berkefeld filter), he had tried using glass plates about 2 feet across, which enabled a much larger quantity to be worked upon.

He had obtained better results with Elsner's method than with the other published methods, and if any likely colonies were taken from Elsner's medium and put into milk, those that caused curdling could be rejected as certainly not being typhoid; there would not then be such a large number of colonies that it would be impossible to deal with them.

When agar was soaked overnight it dissolved in hot broth in about half an hour.

When organisms were cultured through several generations, they changed considerably in size, so that the size of the organisms could not be regarded as affording very conclusive evidence. The number of flagella was also liable to alteration. It was well known, for instance, that the number of flagella possessed by the cholera bacillus was reduced, after prolonged subculture, from three or four to one or two only.

Dr. Pakes said he would like to mention that the substitution of powdered agar for the ordinary agar rendered the making of this medium extremely simple. They had used it in the laboratory at Guy's Hospital for the last two and a half years, and would certainly not like to go back to the old kind.

Mr. Stoddart said he thought he had been treated very kindly in the discussion, most of the observations having been rather in his support than otherwise.

With reference to the remark of Dr. Pakes that a mixed growth of typhoid and coli, unless the typhoid was largely in excess to start with, would result in the suppression of the typhoid, he thought that, with a slight verbal change, it would be found that this was exactly what he himself had said, which was to the effect that when any of several of the bacilli referred to in the paper, and which would commonly be called colon bacilli, were grown in broth with typhoid, the latter was more or less completely suppressed, and could not be found by subsequent plate culture. The organisms which interfered in this way were generally those which behaved like typhoid in halo culture.

This, however, was not the case with other coli-like forms, or with the true *B. coli* itself, as received from Dr. Escherich. When typhoid was grown simultaneously with the last, there was no difficulty in recovering it by the halo culture.

He had never made any exact determinations of acidity with a view to distinction. It did not seem to him that a quantitative determination would confer any additional powers of discrimination, nor had he been aware that it was made use of in discriminating between the various members of this group of organisms. It was, however, a very interesting point, and he would certainly try if it would throw any light on the subject.

With regard to the matter of temperature, he could only say that he had made the medium exactly in the way he had described, and had kept the incubator at 35° C. The medium did not melt at that temperature, though it became so soft as to

shift if the plate was moved. It was necessary that it should be as soft as this in order to get the distinctive growth.

Those experiments on the pathogenicity of organisms which seemed so easy to make in London were practically out of the power of persons living in the provinces, where there was a great deal of opposition to anything in the nature of experiments on living animals, and this was the case in Bristol probably more than anywhere else. Any bacteriological investigation made in Bristol must necessarily be defective for this reason. He could not help this, but to attempt to make such experiments would be almost to risk one's life He admitted that it was a very grave defect in his paper, but he did not think it ought to altogether prevent his investigating this subject.

As to the effects of different surroundings, it was perfectly true that the same organism grown under widely different circumstances would change very considerably in its behaviour, but he hardly thought that this would apply to the present cases.

These thirteen organisms were obtained from different sources. C_{11} was from a water that nobody could complain of, either from an inspection of its source or from its chemical analysis. Some of the others were from fæcal matter. Nos. 2, 3, 4, and 5 were all given to him in pure culture as typical colon bacilli; but all had been carried through very numerous generations, in some cases for years, and yet they maintained the same distinctions.

The numbers of flagella given in the table represented the maximum numbers observable in a great many preparations. He did not mean to say that the difference between, say, 5 and 8 flagella was sufficient for positive discrimination, but, broadly speaking, he could draw a definite distinction between an organism which with some difficulty gave a single flagellum and another organism which showed an abundance of flagella.

The use of large dishes appeared to be an admirable modification. He had never himself adopted it, but it seemed to him a very proper modification of the plate process. However, with the method described in the paper, concentration to a small bulk was of course an advantage.

As to the practical suggestions which had been offered for obtaining good cultivation media, he could only express his gratitude for these, and say that he would certainly take the first opportunity he could of putting them into practice.

The PRESIDENT, in asking the meeting to accord its hearty thanks to Mr. Stoddart, said that no doubt all those present would like to see the preparations which Mr. Stoddart had kindly brought up. They were indebted to Mr. Ling and to Messrs. Watson for supplying several microscopes with excellent lenses; and with the aid of these Mr. Stoddart would have some demonstrations to make for those who would like to see them. He would also ask the meeting to return its thanks to the visitors for their contributions to the discussion.

ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

FOOD AND DRUGS ANALYSIS.

The Determination of Aloin in Aloes. G. L. Schafer. (*Pharm. Zeit.*, 1897, 95; through *Jour. Pharm. Chim.*, 1897, 296.)—The method is based on the fact that aloin in ammoniacal solution forms compounds with the alkaline earths, which are but little soluble, and from which the aloin can be recovered on treatment with an acid.

Fifty grammes of aloes are treated with 300 c.c. of boiling water containing a few drops of hydrochloric acid, and when cold the solution is separated from the resin. Fifty c.c. of 20 per cent. ammonia and 15 grammes of calcium chloride in 30 c.c. of water are then added, and the whole well shaken. After fifteen minutes the precipitate is separated, and after being pressed is triturated in a mortar with a slight excess of hydrochloric acid. The aloin and calcium chloride are dissolved in as little boiling water as possible, and filtered. On cooling the aloin separates in crystals.

The amount of aloin found by the author in different varieties of commercial aloes varied between 15 and 30 per cent. C. A. M.

ORGANIC ANALYSIS.

Determination of Fat in Oil-cakes. A. Bilteryst. (*Bull. de l'Ass. belge*, 1897, x., 406-412.)—In this paper it is shown that carbon tetrachloride may be advantageously substituted for ether in extracting the fat from oil-cakes. The extraction may either be made in a Soxhlet's tube in the ordinary manner, or the pulverized cake may be extracted in the cold for twenty-four hours, and the extract determined in an aliquot portion of the solvent. In the former case preliminary drying of the substance at 100° C. is unnecessary, since carbon tetrachloride is not miscible with water; the risk of the solvent taking fire is obviated, and the extraction requires less time.

For the cold digestion, 20 grammes of the cake are mixed with 100 c.c. of carbon tetrachloride measured at 15° C., and left for twenty-four hours, an occasional shake being given. Fifty c.c. of the solution are filtered, evaporated on the water-bath, and the residue dried to constant weight. To obtain the amount in 10 grammes of the oil-cake, a correction is necessary for the volume occupied by the fat. This is made by dissolving the residue in 50 c.c. of carbon tetrachloride, noting the increase in volume, and calculating the result by proportion.

The following examples are taken from a long table:

	Extraction with Ether. Fat per cent.	Extraction with CCl_4 Hot. Fat per cent.	Extraction with CCl_4 Cold. Fat per cent.
Oil-cake (earth-nut)	7·94	7·95	7·92
,, ,, ...	9·07	9·10	9·12
,, (colza)...	10·45	10·40	10·43
,, (sesame)	14·80	14·80	14·75

C. A. M.

Examination of Beeswax. **R. Henriques.** (*Zeit. für öffent. Chemie*, iii., 66.)—In the preceding number of the *Zeit. für öffent. Chemie*, Woy drew attention to the difficulty of saponifying waxes by boiling with alcoholic $\frac{N}{2}$ solution, owing to the saponifiable portion being protected from the action of the alkali by the portion not saponifiable.

The author had already pointed out this difficulty in his paper on cold saponification (*Zeit. für angew. Chemie*, 1896, 221). He specially recommended his method of cold saponification as suitable in the case of beeswax, since the difficulty referred to is entirely obviated by the wax being dissolved in benzene prior to the addition of the alkali. Moreover, by this method saponification is complete at the ordinary temperature of the room, and therefore all danger of alteration in the volumetric solution is avoided. The author has had many opportunities of proving the accuracy of the method when applied to beeswax, both pure and adulterated, and he believes it would prove the most convenient method in many other cases, as, for instance, for wool-fat and resins. (See also ANALYST, vol. xxi., pp. 67 and 192.) H. H. B. S.

Adulterated Japan Wax. **C. H. La Wall.** (*Am. Journ. Pharm.*, 1897, lxix., 18-21.)—It is stated by the author that Japan wax is now being extensively adulterated in America with starchy material, to the extent of from 20 to 25 per cent. This was first detected in October, and the admixture has, so far as is known, always been made after the importation.

The appearance of the adulterated product is different from that of the genuine wax, being in most cases free from the network of cracks usually noticed on the surface of Japan wax. The presence of starch is readily detected by fracturing a cake and testing the freshly-exposed surface with iodine. The averages of the constants of four adulterated samples were: specific gravity, 1·0653; melting-point, 52° C.; saponification number, 173·28. From pure samples examined at the same time the figures were: specific gravity, 0·980; melting-point, 54° C.; saponification number, 220·98. The amount of foreign matter indicated by the lowering of the saponification number was calculated to be 21·24 per cent. The starch was also directly estimated by extracting the wax with chloroform, and washing the residual starch with ether and drying it at 100°. The quantity thus found was 23·42 per cent. The nature of the starch employed was not uniform. Under the microscope it was unmistakably identified as corn starch in one case, but in others there was some doubt as to its identity. C. A. M.

On the Chemical Examination of Cacao Butter. **F. Filsinger.** (*Zeit. für öffent. Chemie*, iii., 34.)—Cacao butter is found adulterated with cocoa-nut-oil and the surrogate fat which is prepared from it, with fine preparations of tallow, and with fatty oils principally sesame-oil. Paraffin and marrow-fat are no longer used for the purpose. The following tests, in addition to those of taste and smell, may be applied:

1. The iodine number.
2. The saponification number.
3. The acid number.
4. The melting-point.

Eventually, also, the refraction and the ether and ether-alcohol tests of Bjorklund and Filsinger. The iodine number, without doubt, throws the most light upon the question of adulteration. The number varies from 33·4 to 37·5. The addition of cocoa-nut-oil or its preparations (iodine number 4 to 8), or of preparations of tallow and sesame-oil (iodine numbers 40 to 45 and 108 to 110), respectively lower or raise the iodine number.

The saponification numbers of cacao butter and of cocoa-nut-oil are 195 and 250 respectively. It has not long been known that the acid number may rise as high as 40° to 50° Burstyn. When, however, this is the case, the iodine number is found to rise with it.

It is seldom the case that striking alterations of the melting-point arise through the admixture of foreign fats; even sesame-oil, in quantities usually added, occasions but a slight reduction. The melting-point of pure cacao butter varies from 32·0° to 33·6°. Information regarding refraction is as yet incomplete. Observations made so far at 35° C. give 50.

The purely empirical tests with ether and ether-alcohol, which were formerly relied upon, have now given place to more exact scientific methods. They are, however, still useful at times, as corroborative evidence in doubtful cases.

H. H. B. S.

Detection of Cholesterin in Fats. A. Forster and R. Riechelmann. (*Zeit. für öffent. Chemie*, iii., 10.)—Salowsky's method for the separation of cholesterin from fats has several drawbacks. It requires a considerable length of time for its execution; it takes a comparatively large quantity of ether (500 c.c. for 10 grammes of fat); the separation of the fluids is slow and often imperfect, and the saponification never complete.

By the method of Kossel and Obermüller, the separation of the soap is slow and incomplete, and the ethereal solution yields on cooling a crystallizable oil capable of further saponification.

The authors recommend the following process: 50 grammes of fat are twice boiled for about five minutes with 75 c.c. of alcohol of 95 to 96 per cent. in a flask with a reflux condenser attached, the flask meanwhile being well shaken. The alcohol is then cooled, to allow of the solidification of the fat, which is filtered off. The filtrate is mixed with 15 c.c. of a 50 per cent. soda solution, and boiled on the water-bath in a flask, with a condensation-tube attached, until about one-fourth of the alcohol has evaporated. The fluid is then poured into a porcelain basin, evaporated nearly to dryness, and the residue transferred to a shaking cylinder and shaken up with ether. The ethereal solution is evaporated to dryness, the residue treated with a little ether, filtered, evaporated, and the residue recrystallized from 95 per cent. alcohol.

H. H. B. S.

The Caffeine Compound in Kola. J. W. T. Knox and A. B. Prescott. (*Jour. Amer. Chem. Soc.*, 1897, xix., 63-90.)—In the authors' opinion none of the methods for the assay of kola have proved satisfactory, and the discrepancies in various analyses are to be attributed (1) to incomplete liberation of the alkaloids from their combination, and (2) to the caffeine being weighed in a state of greater or less purity, according to the method employed.

They consider that Gomberg's volumetric process of estimating caffeine (ANALYST, xxi., 193) is far more reliable than gravimetric methods in the analyses of kola, and in the following manner have obtained uniform and concordant results:

Preparation of Sample.—A sufficient quantity of the fresh sample is cut into very thin slices, which are allowed to fall into boiling alcohol, and after a few minutes' boiling removed and allowed to dry spontaneously on glass plates. The alcoholic solution is distilled to a syrup under reduced pressure (to avoid decomposition of the caffeine compound) and poured over the drying slices. When dry the drug is powdered in a mortar and preserved in stoppered bottles. As thus prepared the powder correctly represents the fresh seed without the water, and possibly a trifle of the volatile oil.

Free Alkaloids.—Five grammes of the powder are extracted for six hours with chloroform. To the residue, after evaporation of the chloroform, 30 c.c. of 1 per cent. hydrochloric acid are added, the liquid filtered to remove fat, and the filter washed with hot water. The united filtrate and washings (about 75 c.c.) are concentrated to 10-15 c.c., transferred to a graduated cylinder, and made up to 30 c.c. Thirty c.c. of standard Wagner's solution (1 c.c. = 0·00485 caffeine) are then run in from a burette, and the cylinder is well shaken. After standing for about five minutes the liquid is filtered through asbestos, an aliquot portion titrated with standard thiosulphate, and the amount of iodine absorbed by the alkaloids calculated into caffeine.

Combined Alkaloids.—The drug, after complete extraction with chloroform, is again extracted with 90 per cent. alcohol, two or three hours being usually sufficient. This alcoholic extract may then either be evaporated in a weighed basin, and the nitrogen contained in a small amount determined by combustion and calculated into caffeine, or to the hot alcoholic solution an excess of freshly-precipitated lead hydroxide may be added, the whole digested on the water-bath until the supernatant liquid is colorless, then mixed with sand, evaporated to dryness, the residue extracted with chloroform, and the caffeine determined volumetrically as before.

The following table gives the results obtained by this method of assay:

Sample.	Fresh Kola.				Dried Kola.		
	Moisture per cent.	Free Alkaloids per cent.	Combined Alkaloids per cent.	Total.	Free Alkaloids per cent.	Combined Alkaloids per cent.	Total.
No. 1. Dried kola, mixed—I. ...	6·16	—	—	—	1·859	1·783	3·642
,, ,, II. ...	—	—	—	—	1·828	1·836	3·664
Average	—	—	—	—	1·843	1·809	3·652
No. 2. Fresh kola, red and white seeds. Average ...	53·9	0·534	0·884	1·418	1·158	1·922	3·080
No. 3. Fresh kola, red and white seeds, very mouldy. Average	53·9	0·569	0·854	1·423	1·235	1·854	3·089
No. 4. Fresh kola, white seeds. Average	51·2	0·578	1·018	1·596	1·186	2·085	3·271
No. 5. Fresh kola, red seeds. Average	57·3	0·478	0·693	1·171	1·120	1·625	2·745

The method of Dohme and Engelhardt (*Am. Drugg.*, 1896, 12), which consists in boiling the dried and powdered drug for two hours in 30 per cent. alcohol, evaporating with sand and magnesia, extracting with chloroform, and weighing the dried residue, does not give reliable results. Thus, No. 1 in the preceding table when thus assayed yielded only (1) 2·04 and (2) 1·93 per cent. of caffeine, and even this, when titrated with Wagner's solution, was found to be not pure. But the principal source of error is shown to be owing to the fact that 30 per cent. alcohol does not completely remove the caffeine from its combination.

The Glucoside of Kola.—It is generally stated that kola contains a glucoside, *kolanin*, which is readily decomposable by heat, moisture, dilute acids, the ferment of kola, and by diastase, and which yields on decomposition caffeine, glucose, and ikola-red. But the authors consider that it is highly probable that this substance is really a mixture of tannates of caffeine and theobromine. They have prepared an artificial tannate of caffeine which closely corresponds both in ultimate composition and in chemical and physical characteristics with the natural kolanin.

Experiments with diastase and the kola ferment showed that any liberation of caffeine which occurred was due to the action of moisture and heat. Moreover, sterilization, although checking the formation of the coloured body, kola-red, did not interfere with the liberation of the alkaloids. Hence, the authors conclude that there is no evidence to show that kola-red and caffeine are joint products of the one hydrolysis of a glucoside, though such has been the conclusion of previous investigators.

Estimation of Theobromine.—The method of Kunze (ANALYST, xix., 94), with slight modifications, gives very satisfactory results. The ratio of theobromine to caffeine is fairly constant, and in a typical case the amount found in the free alkaloids was 1·48 per cent., and in the combined alkaloids 1·51 per cent.

Free Tannin.—For this estimation the authors employed a modification of the process given by Allen (*Comm. Organ. Anal.*, iii., part i., 76). The drug was exhausted with 95 per cent. alcohol, and the solution distilled *in vacuo* to a syrup, and then washed with water. The insoluble matter was removed and the clear solution fractionally precipitated with lead acetate (or hydroxide), the first and last portions of lead tannate being rejected. The lead tannate was well washed, suspended in alcohol, and decomposed with sulphuretted hydrogen. After filtration the alcoholic solution of tannin was distilled *in vacuo* to a syrupy consistence, and the evaporation completed in a vacuum desiccator over sulphuric acid. The tannin thus obtained was light red to reddish-brown, and behaved as other tannins do towards iron salts, gelatin, alkaloids, etc. It was a glucosidal body, yielding on hydrolysis with mineral acids glucose and a dark brown substance insoluble in water or alcohol, and containing carbon 69·20 per cent. and hydrogen 6·70 per cent.

Combined Tannin.—The tannin, in combination with the caffeine in kola, was separated by means of lead hydroxide. It agreed in appearance, composition, and properties with the free tannin, being also a glucosidal substance.

See also abstracts on kola, ANALYST, xx., 42, and xxi., 265 and 292.

C. A. M.

Valuation of Raw and Manufactured Caoutchouc. R. Henriques. (*Zeit. für öffent. Chemie*, iii., 37.)—Raw caoutchouc contains water; inorganic substances, as sand, stone, clay, etc.; organic impurities, accidentally introduced, as leaves and twigs; gum resins from other plants added by way of adulteration; and the so-called caoutchouc resin, which is distinguished from the true caoutchouc substance by its ready solubility in ethylic and methylic alcohol and in acetone. That which remains after the separation of these constituents is the pure caoutchouc substance, upon which the value of the commercial product depends.

The analysis of raw caoutchouc is carried out as follows: A weighed portion (50 to 100 grammes) is macerated in water in a small roller apparatus—similar to the kneading and washing machine used in indiarubber works—by which the mechanical impurities are eliminated. The resulting rubber is dried at 80° to 90°, and weighed. If a proper macerating apparatus is not at the operator's disposal, the mechanical impurities may be got rid of by the aid of water and a pair of scissors. Five to 10 grammes of the washed and dried rubber are then digested for two to three hours in acetone in a Soxhlet apparatus, to effect the extraction of the caoutchouc resin, the residue being then quickly dried and weighed. The determination of the ash completes the analysis, the percentage of true caoutchouc being obtained by difference.

The principal constituents of manufactured rubber are:

1. Added substances of the most divers description, such as lead oxide, heavy spar, gypsum, zinc oxide and carbonate, chalk, potash, magnesia and magnesium carbonate, golden antimony sulphide, ferric oxide, kieselguhr, asbestos, powdered glass, pumice-stone, etc.
2. Caoutchouc resin, soluble in alkalies.
3. Caoutchouc surrogate, asphalt and gum resins.
4. Free sulphur.
5. Caoutchouc resin in the insoluble form.
6. Combined sulphur, as vulcanite.
7. True caoutchouc substance.

The following is the procedure for the analysis of manufactured caoutchouc: The sample is reduced to a fine powder by the aid of a sharp file. Five grammes are then boiled for from three to four hours in a 3 per cent. to 4 per cent. alcoholic solution of sodium hydrate, which effects the solution of the free sulphur, resin, caoutchouc surrogate and the soluble caoutchouc resin, in addition to which, of course, many inorganic constituents may be attacked and partly dissolved. The solution is then filtered while still boiling, and washed with boiling alcohol. If the presence of asphalt be suspected, the residue is treated with cold nitrobenzol; otherwise this operation may be omitted. The residue is then washed into a beaker and boiled several times, first with water and afterwards with hydrochloric acid, the washings each time being decanted through the same filter. The residue is then all brought on to the filter, washed with hot water, pressed, dried at 80° to 100°, and weighed. The drying must not be continued longer than necessary, as the dry caoutchouc gradually takes up oxygen. The residue now contains the caoutchouc substance, the combined sulphur, insoluble inorganic substances, such as silicates, barium sulphate, etc., a very trifling quantity of the caoutchouc resin, and a little

carbonaceous matter added for coloring purposes. If the ash and the sulphur be now determined and their amounts deducted from the residue, the remainder will be the true caoutchouc substance.

H. H. B. S.

INORGANIC ANALYSIS.

The Application of Nitroso-β-Naphthol to Inorganic Analysis. R. Burgass. (*Zeit. angew. Chem.*, 1896, 596-601.)—When in solution as chlorides or sulphates, iron and cobalt can be precipitated together by an acetic acid solution of nitroso-β-naphthol, the oxides weighed after ignition, then dissolved in hydrochloric acid, and the iron determined in the usual manner. The nitroso-naphthol is dissolved in 50 per cent. acetic acid, and in making the determination 10 to 20 c.c. of acetic acid must also be added to the solution of the metals, which has been previously heated with 2 or 3 c.c. of hydrochloric acid and allowed to cool to 95° C. The precipitate is washed with hot water (not too much) containing a little hydrochloric acid, and then with water alone, and ignited without previous drying in a capacious platinum crucible.

Copper is also precipitated quantitatively, while the following metals remain in solution: mercury, nickel, chromium, manganese, lead, zinc, aluminium, cadmium, magnesium, calcium, beryllium, antimony, and arsenic.

Silver, tin, and bismuth are only partially precipitated, and must therefore be removed before adding the nitroso-naphthol. In order to prevent any antimony being precipitated as oxychloride, 10 to 15 c.c. of a solution of tartaric acid (1 : 5) should be added. The antimony cannot be determined directly as sulphide in the filtrate, since the latter always contains some nitroso-naphthol. The sulphide should therefore be treated with fuming nitric acid, and the antimony determined as oxide by igniting in a porcelain crucible.

Similarly, arsenic cannot be directly determined in the filtrate, which should be oxidized with potassium chlorate and hydrochloric acid, and the arsenic then estimated as magnesium pyro-arseriate.

In the case of iron, phosphoric acid is simultaneously precipitated, sometimes almost quantitatively, and hence interferes with the determination; but it does not affect the estimation of cobalt or copper. The determination of all three is vitiated by the presence of tungstic or molybdic acids.

In order to obtain correct results by this method, it is necessary to take not more than 0·3 gramme of the substance to be precipitated, and to have a sufficient quantity of free acetic acid present. C. A. M.

Formaldehyde as a Reducing Agent in Analysis. B. Grützner. (*Arch. Pharm.*, 1896, ccxxxiv., 634; through *Chem. Zeit. Rep.*, 1896, 314.)—On acidifying a solution of potassium chlorate containing silver nitrate and formalin with nitric acid, the chlorine separates quantitatively as silver chloride, especially on warming; and the reaction may be employed gravimetrically or volumetrically for estimating either the formalin or chloric acid. Mixtures of chlorides and chlorates may be titrated

with silver to give the chloride, and then reduced with formalin and nitric acid and again titrated to obtain the chlorate. Bromates behave in a similar manner, but it is necessary to heat the liquid for two or three hours on the water-bath. Periodic and perchloric acids are only slightly reduced, and iodic acid not at all.

F. H. L.

Preparation of Pure Oxalic Acid. R. Riechelman. (*Zeit. für öffentl. Chemie*, iii., 13.)—For the preparation of chemically-pure oxalic acid for use in standardizing volumetric solutions, it is recommended to recrystallize the commercial product, first from ether and afterwards from water. For expediting the solution of the oxalic acid in ether, the use of Soxhlet's apparatus is advised. H. H. B. S.

On the Constancy of Standard Solutions of Permanganate when Protected with a Layer of Vaseline Oil. Meineke and Schroeder. (*Zeit. für öffentl. Chemie*, iii., 5.)—Although solutions of permanganate keep well when prepared from the pure crystallized salt, yet when stored in large vessels with syphon connections to burettes there is always a danger of alteration through loss of water by evaporation. As a protection against this, the authors propose a layer of vaseline oil floated upon the surface of the solution, and they have carried out a series of experiments to ascertain if it exercised any reducing effect upon permanganate.

Experiments were made with the oil in its natural state, as well as after purification by treatment with permanganate in alkaline, neutral, and acid solutions.

The authors conclude that while the results obtained with the purified oil do not justify the trouble bestowed upon it, the reducing action of the oil in its natural state is so very slight that it may well be disregarded. H. H. B. S.

Detection and Estimation of Perchlorate in Nitrate of Soda. 1. Erck (*Chem. Zeit.*, 1897, xxi., 10); 2. F. Winteler (*loc. cit.*, p. 75.)—It has recently been stated by Sjollema that the harm done to certain rye crops by nitrate of soda was caused by the presence of sodium perchlorate in the material, and Erck therefore suggests the following process for its examination: 100 grammes are dissolved in 80 c.c. of water, 7 c.c. of 1·4 nitric acid added, the mixture warmed, 8 c.c. of alcohol (92° Tr.) run in, and the whole boiled for about five minutes till the reaction is complete. All the chlorine from the chlorides and chlorates will be driven off, and if the solution is made alkaline with pure sodium carbonate, evaporated to dryness and ignited, after acidifying again with nitric acid, the chlorine from any perchlorates originally present may be detected or estimated with silver in the usual manner. Should the sample contain more than 0·3 per cent. of sodium chloride, it may be necessary to employ larger quantities of acid and alcohol; but this point may be determined by testing a small portion of the liquid before the sodium carbonate is added.

Winteler objects that this process is not adapted for quantitative work, since loss of perchlorate chlorine may occur, through simple volatilization, through decomposition of the perchlorate by the chloride formed on heating (as shown by Gooch and

Kreider), and through the action of the nitric oxide evolved in the first operation. He remarks that perchlorates and chlorates are completely reduced by heating with fuming nitric acid in a sealed tube for five hours at 230°, and that in a mixture of these salts the chlorate may be decomposed by evaporation with strong HCl. Tests on potassium perchlorate, chlorate, and "pure" nitrate (contaminated with $KClO_4$) have shown the process to be perfectly reliable; and it should be equally available for treating nitrate of soda. F. H. L.

Solubility of Phosphates in Citric Acid and Ammonium Citrate. O. Foerster. (*Chem. Zeit.*, 1896, xx., 1020.)—In the following table the author has collected the results obtained on treatment with (1) ordinary Wagner solution, and (2) 1·4 per cent. free citric acid, of certain definite phosphates and also those few samples of basic slag which, out of a large number examined, have shown an appreciably different behaviour towards the two reagents. The expression "gently ignited" means that the substance was only heated sufficiently to drive off the water of hydration; and the figures given by the basic calcium phosphate explain in part the differences found by previous investigators (*cf.* ANALYST, xxi., 81 and 166). The reason of the abnormal action of slags 7 and 8 is not obvious; and Foerster is experimenting further on the subject, but it probably depends on certain double ammonium salts, formed when Wagner's liquid is employed, acting as solvents on some of the otherwise insoluble phosphates. The author remarks that he has met with several slags containing so little silica that scarcely half of the actually "available" acid was extracted by ammonium citrate.

Sample Examined.	Total Phosphoric Acid per cent.	Percentage of Total Phosphoric Acid dissolved by	
		Ammonium Citrate.	Citric Acid.
$Ca_3P_2O_8$ strongly ignited	45·81	43·8	57·6
$(Ca_3P_2O_8)_3CaO$ strongly ignited	43·20	7·4	20·2
$FePO_4$ strongly ignited	47·02	—	0·3
,, gently ignited	47·02	—	0·3
$AlPO_4$ strongly ignited	58·20	—	—
,, gently ignited	58·20	1·7	8·9
Slag No. 1	20·69	38·3	44·1
,, 2	19·58	52·6	58·3
,, 3	16·81	38·5	42·4
,, 4	18·82	66·4	69·6
,, 5	17·63	39·6	42·8
,, 6	18·66	68·0	70·1
,, 7	13·94	95·3	91·1
,, 8	17·97	89·4	83·6

F. H. L.

Determination of the Citrate-Soluble Phosphoric Acid in Thomas Phosphate Powder. O. Bötcher. (*Chem. Zeit.*, 1897, xxi., 168.)—The author seeks to improve upon the German official method (Wagner's) for the determination of citrate-

soluble phosphoric acid in Thomas phosphate powder. Wagner's method is open to certain objections, especially that the solutions required are different from those used for the ordinary determination of phosphoric acid in manures, which is both inconvenient and a possible source of mistakes, and that the final precipitate of magnesium ammonium phosphate is not always free from silica.

The following method was first tried: To 50 c.c. of the citrate solution of Thomas phosphate prepared according to Wagner's directions a teaspoonful of sodium nitrate was added, and the whole evaporated to dryness and ignited. The residue was moistened with hydrochloric acid and dried for two hours at 120° C. to ensure complete separation of the silica. It was then dissolved in hydrochloric acid by warming, the solution diluted with water, filtered through a small filter, the filter washed with hot water, and the phosphoric acid determined in the filtrate by the citrate method (precipitation by magnesia mixture) in the usual way. The results agreed exactly with those obtained by Wagner's method. The author did not, however, consider this method completely satisfactory, as, though simpler than Wagner's owing to the precipitation by molybdenum and the use of special solutions being avoided, it was still a somewhat intricate process.

The following method was then tried: 50 c.c. of Wagner's citrate solution of Thomas phosphate was taken, and the phosphoric acid precipitated by the citrate (magnesia) method in the usual way. The precipitate was filtered off, washed with a 5 per cent. ammonia solution, and, together with the filter, ignited while still moist, in a platinum crucible. The residue was then dissolved in warm hydrochloric acid, the solution diluted with water, filtered, the filter washed with hot water, and the phosphoric acid determined in the filtrate by the citrate method, as before. The author finds that this method yields satisfactory results. The pyrophosphate is completely reconverted into orthophosphate by the treatment with warm hydrochloric acid, and the resulting magnesium ammonium phosphate is pure and practically free from silica.

H. H. B. S.

The Estimation of Potassium. A. Mercier. (*Bull. de l'Ass. belge*, 1897, x., 403-405.)—The author has proved that mercurous chloride can replace sodium formate for the reduction of potassium platinic chloride, and on this fact has based the following process: Five or ten grammes of the sample (according to its richness in potassium) are digested for an hour with 500 c.c. of cold water and filtered; 25 or 50 c.c. of the filtrate are evaporated to dryness on the water-bath after the addition of 1 c.c. of hydrochloric acid. The residue is gently ignited to expel ammonium salts, taken up in very dilute hydrochloric acid, and filtered if necessary. Ten c.c. of platinic chloride solution (10 per cent.) are then added, and the liquid evaporated on the water-bath to a syrupy consistency, cooled, and extracted with 50 c.c. of 85 per cent. alcohol + 5 c.c. of ether. The double salt is well washed on the filter with the same alcohol-ether, and then dissolved in boiling water.

The aqueous solution is brought to the boil, and mercurous chloride added little by little until it is deposited on the bottom of the beaker. In the majority of cases 2 grammes are sufficient. After five minutes' boiling the platinum is allowed to settle

to make sure that the reduction is complete, then 1 or 2 c.c. of hydrochloric acid are added and the liquid boiled again. When cold the liquid is filtered, and the platinum, which is far more dense than that obtained with sodium formate, is well washed with boiling water and ignited without previous drying in a platinum crucible. The weight of platinum obtained, multiplied by 0·4835, gives the amount of potassium in terms of K_2O. The results obtained by this method, which are tabulated at the end of the original paper, are in close agreement with those required by theory. C. A. M.

A Modification of the Schweitzer-Lungwitz Method of estimating Potassium. A. Mayer. (*Zeit. anal. Chem.*, 1897, xxxvi., 159-163.)—The method of Schweitzer and Lungwitz consists in first separating the sulphates by means of a solution of barium oxalate in hydrochloric acid, oxidizing any iron present with hydrogen peroxide, and then adding ammonia to precipitate the alkaline earths as oxalates, and iron and aluminium as hydrates. Since barium oxalate is only soluble in hydrochloric acid to the extent of 1·7 per cent. in the cold, over 800 c.c. of the solution must be used in the case of superphosphates where 15 grammes of barium oxalate are required.

In the author's modification barium chloride and oxalic acid are substituted for barium oxalate in hydrochloric acid, a course which was taken into consideration by Schweitzer and Lungwitz, and rejected by them. Twenty grammes of the substance, ash, manure, etc., are boiled for half an hour with water, the liquid cooled, made up to 500 c.c., and filtered. Fifty c.c. of the filtrate are heated, and normal barium chloride solution added from a burette, till no more precipitate is produced. An equivalent volume of normal oxalic acid solution is added, the liquid boiled for twenty minutes, and made alkaline with ammonia. After cooling, it is made up to 100 c.c., allowed to settle, and filtered. Fifty c.c. of the filtrate are evaporated in a platinum basin, the residue ignited at as low a temperature as possible to expel ammoniacal salts, dissolved in water, filtered, and the potassium precipitated by adding 10 c.c. of platinic chloride solution (containing 1 gramme of platinum in 10 c.c., and which is free from hydrochloric and nitric acids), evaporating almost to dryness. When cool, alcohol (80 per cent. by vol.) is added, and after being stirred for some time and allowed to stand, the precipitate is filtered off and dried at 120° C.

The following table shows the agreement between the results obtained by this method and those given by the ordinary method:

	Schweitzer-Lungwitz Modification. Potassium per Cent.	Ordinary Method. Potassium per Cent.
Mixture of kainite and superphosphate	7·1	7·0
Ash from iron furnace, No. 176	11·9	11·9
,, ,, ,, 187	11·1	11·1 to 11·5 (2 analysts)
,, ,, ,, 189	9·6	9·6
Potassium superphosphate ...	7·4	7·3 to 7·4 (2 analysts)

It is essential to boil the liquid for the full twenty minutes after adding the oxalic acid, or the results obtained will be too low. C. A. M.

A Rapid Method for the Determination of Silicon in Silico-Spiegel and Ferro-Silicon. C. B. Murray and G. P. Maury. (*Jour. Amer. Chem. Soc.*, 1897, xix., 138, 139.)—The authors having tried the "aqua regia" method and the fusion method of Williams (*Trans. Am. Inst. Min.*, xvii., 542) for the determination of silicon in silico-spiegel, and having found the former almost impracticable and the latter too tedious, hit upon the following method:

The sample is finely ground in a diamond mortar so as to pass through a bolting-cloth sieve. To half a gramme in a porcelain or platinum dish are added 50 c.c. of water, 10 c.c. of hydrochloric acid (specific gravity 1·20), and 12 c.c. of sulphuric acid (1 part of acid, specific gravity 1·84, to 3 parts of water), and the basin is heated until fumes of sulphuric acid are given off. When cool, about 10 c.c. of hydrochloric acid are added, heat applied, then about 75 c.c. of water added, and the liquid boiled. If there is any effervescence when the boiling ceases the liquid must be evaporated until fumes of sulphuric acid are again given off, and the residue taken up as before. The liquid is then filtered, the residue washed thoroughly with hydrochloric acid (1 : 1) and hot water, ignited in a platinum crucible and weighed. A few drops of sulphuric acid are added, and sufficient hydrofluoric acid to dissolve the silica, and the contents of the crucible are evaporated to dryness, heated to decompose the sulphates, cooled and weighed. The difference in the two weights gives the amount of silica which can be calculated to silicon.

The following are some of the results obtained by this method, which occupies about thirty minutes, compared with those yielded by the fusion method (six to eight hours):

	New Method.	Fusion Method.
1.	12·08	12·01
2.	12·37	12·25
3.	12·09	12·08
4.	13·46	13·40
5.	9·05	9·03

C. A. M.

APPARATUS.

A New Form of Specific Gravity Bottle. J. C. Boot. (*Jour. Amer. Chem. Soc.*, 1897, xix., 61, 62.)—An objection to the ordinary form of specific gravity bottle is that when the temperature of the room is much higher than the normal temperature, the liquid is continually running out of the capillary tube in the stopper during the drying and weighing. This is obviated in the bottle devised by the author, which has double walls, and in which the space between these two walls is exhausted as completely as possible.

The apparatus can be obtained from Christ. Kob and Co., Stützerbach, Germany; and from Eimer and Amend, New York. C. A. M.

THE ANALYST.

JUNE, 1897.

PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS.

The monthly meeting of the Society was held on Wednesday evening, May 5, at the Chemical Society's Rooms, Burlington House, the President (Dr. Bernard Dyer) occupying the chair.

The minutes of the previous meeting were read and confirmed.

Mr, Henry Bradford, assistant to Mr. E. G. Clayton, was nominated by the Council for election as an Associate of the Society.

The following papers were read: "The Value of the Nitrogen Factor in the Analysis of Decomposed Milks," by Alfred Smetham and J. B. Ashworth; "Notes on the Influence of Boric Acid upon the Action of Digestive Ferments," by R. A. Cripps.

ON COPPER IN PEAS.

By R. Bodmer and C. G. Moor, M.A.

(*Read at the Meeting, March* 3, 1897.)

During the last few years there have been several prosecutions for the sale of preserved peas containing copper. Three cases will be within the recollection of many of our members—the Bristol case, the Edmonton case, and the case of Grist *v.* Summers, at Southwark.

In the Bristol case the magistrates did not convict, although they expressed an opinion that the coppering of peas was objectionable.

In the Edmonton case a conviction was obtained, which was not appealed against.

In the Southwark case an appeal followed, and the matter was fought out very thoroughly. The conviction was confirmed. Shortly after this another case occurred at Southwark Police Court, which resulted in a conviction and a fine of £20 and costs.

We have thought the question of the coppering of peas sufficiently important to bring before the Society a few results of analyses of peas and other vegetables. Many of these were analyses made for private individuals, and not for the purposes of prosecution.

Methods of Analysis.—Tschirch has shown (*Das Kupfer*) that it is most important that the peas should be *completely* incinerated; this has been confirmed by Paul and Cownley and also by our own experiments. If the peas are only charred,

even nitric acid fails to extract all the copper. One hundred grammes of peas, from which the liquor contained in the tin or bottle has been poured off, are incinerated in the muffle at a moderately low temperature. The charred mass is then moistened with nitric acid and completely incinerated. The ash is taken up with nitric acid, water is added, the mixture boiled and filtered. The filtrate is evaporated to dryness, warmed with sulphuric acid till free from nitric acid, and the residue taken up with water. In this solution copper may be determined by several methods:

1. Colorimetric method with sulphuretted hydrogen.
2. ,, ,, ammonia.
3. ,, ,, ferrocyanide.
4. Gravimetric method by electrolytic precipitation.

Of the colorimetric methods, the first gives results somewhat too high. The second method gives very accurate results, if the proportion of copper is not too minute. For very small quantities of copper, the ferrocyanide method is the most accurate. On the whole, we are inclined to believe that the best results are to be obtained by electrolytic deposition of the copper on platinum.

Preserved peas are coloured with copper by heating them for a short time with a solution of copper sulphate, which is then poured off, and the peas are washed to remove all copper salt that has not actually become fixed in the pea itself.

It follows, then, that the brine in which the peas are preserved contains only traces of copper, and hence it is a point of importance to decide whether for purposes of analysis the peas should be ground up with the liquor and the mixture analysed, or whether the liquor should be first poured off and the peas analysed alone. In the analyses made by R. Bodmer the figures represent the copper in the peas alone, while those by C. G. Moor were made on a mixture of the entire contents of the tin. If the division of the sample is made by an inspector, it is easy to see that the three portions may vary very considerably in the proportion of peas and liquid they contain.

PRESERVED PEAS.

Sample.	Description.	Copper in Grains per lb.	Copper as Sulphate.	
1.	Extra fine green peas	0·98	3·87	R. B.
2.	Fine green peas	0·98	3·87	,,
3.	Green peas fins	0·84	3·32	,,
4.	,, ,, ...	0·84	3·32	,,
5.	Green peas moyens	0·42	1·66	,,
6.	Extra fine green peas	1·26	4·97	,,
7.	Green peas mi-fins	1·26	4·97	,,
8.	,, ,, fins	1·50	5·92	,,
9.	,, ,, ,, ...	0·63	2·48	,,
10.	Extra fine green peas	1·51	5·96	,,
11.	Petit pois mi-fins	1·00	3·90	C. G. M.
12.	Petit pois fins	1·70	—	
13.	Petit pois extra fins	1·00	3·90	
14.	Preserved vegetables	0·70	—	
15.	Petit pois fins	0·30	—	
16.	Petit pois surfins	0·50	—	

HARICOT VERTS.

Sample.	Description.	Copper in Grains per lb.	Copper as Sulphate.	
17.	Extra fine haricot verts... ...	0·77	3·04	R. B.
18.	Haricot verts moyen	0·70	2·76	,,
19.	Extra fine haricot verts... ...	1·26	4·97	,,
20.	,, ,, ,, ...	0·42	1·66	,,
	SPINACH.			
21.	Épinard extra	2·24	8·84	,,
	MACEDOINES (MIXED VEGETABLES).			
22.	Macedoines (French)	0·35	1·38	,,
23.	,, de légumes fins ...	0·32	1·26	,,
24.	,, de légumes ...	0·70	2·76	,,
25.	Jardinières	0·52	2·09	,,

In the *Pharmaceutical Journal* of June, 1896, Messrs. Paul and Cownley published a number of figures showing the various amounts of copper found in oysters, cocoa, spirits, and preserved peas. In this paper they quote Lehmann, who "mineralizes" the sample with strong sulphuric acid, and then determines the copper colorimetrically with ammonia or ferrocyanide. They also quote the following results obtained by Vedrödi,* whose determinations were made colorimetrically with sulphuretted hydrogen, and which are given in parts per 10,000:

	Minimum.	Maximum.
Winter wheat	2·5	3·6
Summer wheat	2·5	3·0
Barley	0·1	0·9
Linseed	1·4	1·9
Peas	0·9	1·5
Mustard-seed	0·9	1·0

They conclude that according to Vedrödi some natural vegetables contain more copper than is found in vegetables to which it has been added to preserve their natural colour. Paul and Cownley proceed as follows in estimating copper in peas and other substances: The sample is carbonized in a platinum dish, and extracted with hydrochloric acid; the insoluble residue is ignited with a little nitric acid, hydrochloric acid added, and the resulting mixture is added to the original extract. The solution is then concentrated to about 30 to 40 c.c., placed in a weighed platinum dish, and the copper deposited with pure zinc. If the deposited copper is not pure copper colour, they dissolve it in a little nitric acid and estimate the copper colorimetrically. They give the following figures, which we have averaged. The figures are in parts per 10,000:

		Copper.
Oysters, average of ...	4 samples ...	2·4 nearly
Cocoa, average of ...	3 ,, ...	0·44 ,,
Spirits, average of ...	5 ,, ...	0·03 ,,
Preserved peas, average of	11 ,, ...	1·00 ,,

Of the preserved peas, their highest sample shows 1·44 and their lowest 0·54 parts of copper per 10,000.

* *Cf.* ANALYST, xxi., p. 235.

The following figures have been obtained by one of us on coffee, cocoa, flour, and barley:

	Copper.
Coffee	trace
Cocoa (entire bean) ...	0·5 grain per lb.
Flour...	trace
Barley	trace
Peas (dried)	trace

There is considerable difference of opinion as to whether the coppering of peas renders the article injurious to health. Dr. A. Tschirch has investigated this subject most thoroughly. According to him, the copper introduced in the *re-verdissage* of green peas exists chiefly as phyllocyanate, but if an excessive amount of copper is introduced, a portion is also present as albuminate or leguminate. It is immaterial in which form the copper exists, as from actual experiment on animals he has shown that the insoluble phyllocyanate is, in proportion to the amount of copper it contains, as toxic in its action as such a soluble copper salt as the double tartrate of copper and sodium. The proportion of copper required to fix the green colour of peas is very small. According to Gautier, 18 parts per million—that is, 0·126 grain per lb.—is quite sufficient for the purpose. Tschirch proposes as an extreme limit of the permissible amount of copper, one part of copper in 10,000 parts of the vegetable—that is: 0·7 of a grain per lb.—but considers that 0·35 grain per lb. is amply sufficient for the purposes of the manufacturer. It is therefore evident that the proportion of copper found in many of the samples the analyses of which we have quoted (*Das Kupfer*) is above the amount that he considers safe. As to the question whether copper is a poison, he states on page 105, as the result of experiment on animals and men and the collected cases in medico-legal practice: "We are in a position to answer this question in the affirmative, with the limitation that with men, although it seldom causes death, yet, if administered in neither too large nor too small doses, it is capable of producing more or less severe symptoms of poisoning." This is eminently a matter for the serious consideration of the proposed Committee of Reference in the new Act for regulating the Sale of Foods and Drugs.

In most European countries, with the exception of France, the addition of a copper salt to articles of food is either absolutely prohibited, or it is only permitted under certain specified limits. It is only in this country that it is necessary to argue the point as to whether the addition of a certain proportion of an injurious substance, such as sulphate of copper, does or does not render the article to which it has been added injurious to health.

Discussion.

Dr. Rideal observed that he had recently found traces of copper in a sample of sterilized milk.

Mr. Hehner thought there would be some danger in determining the copper electrolytically. Tin was always present, and this was liable to be deposited and reckoned as copper. He would not be inclined to place much faith in an electrolytic method, unless some security was afforded against the deposition of tin, and this did not seem to be an easy matter. Besides, the colorimetric process was all that could

be desired. No method could lay claim to remarkable accuracy, but the colorimetric process gave results as accurate as any other.

He could not help thinking that the quotation which the authors had taken from Tschirch's book was not sufficiently full. As a matter of fact, Tschirch had actually fed a number of his students on coppered food, and had to give them comparatively large quantities of copper to produce an appreciable effect.

It was true that in most European countries the admixture of copper with foods was prohibited by law. In Germany it was absolutely forbidden, and for some time the public analysts of that country tried to carry out the law, but soon found that it was not possible to do so, and came to the conclusion (in spite of what the law stated) that they would permit 25 milligrammes of copper per kilogramme, or about 1·7 grains per lb.

Mr. CASSAL said he would like to have the opinion of the Society on the point mentioned by the authors with regard to the treatment of samples of this nature before analysis. He had had some correspondence with Mr. Bodmer, from whom he had gained much valuable information on this subject; but he and Mr. Bodmer differed on one very important point, viz., as to whether it was necessary, under the provisions of the Sale of Food and Drugs Act, for a public analyst receiving a sample of this kind to pour off the liquor first, or to treat the sample as a whole, and analyse both liquor and peas. Mr. Bodmer thought that the proper course was to pour off the liquor and analyse the peas, on the ground that it was approximately in that condition that the peas would be eaten. Mr. Moor, on the other hand, took the view which he (Mr. Cassal) himself held, and considered it legally necessary for a public analyst to analyse the sample as submitted to him, liquor and all. He had had some legal opinions on this point, all of which agreed that a public analyst could not, in the present state of the law, do otherwise than analyse the sample as submitted to him, and that it would be sufficient to cause the failure of the case if the public analyst had to admit that prior to analysing the peas he had poured off the liquor. He (Mr. Cassal) admitted that if such a course could be considered permissible he would be very pleased, but, at the same time, he would be very sorry to advise his authorities to go into court in a case of this kind, and then have to admit that he had poured off the liquor before making the analysis, and so done a thing which it was illegal for a public analyst to do. The unfortunate result of being forced to come to this conclusion was that the figures obtained by Mr. Bodmer were rendered of but little use to other public analysts who might be engaged in cases of this kind because the standard created was one created upon Mr. Bodmer's method; and if they were told that the liquor must not be poured off, a fresh standard would be required, and the whole business would have to be refought.

Mr. BODMER said that in the Southwark case the inspector poured off the liquor in the act of dividing the sample. The inspector was asked in court if he had poured the liquor away, and answered in the affirmative, and no objection was taken by either side to this being done. In the case of the other samples, no prosecutions were intended, the analyses having been undertaken for private individuals.

Mr. CASSAL said this seemed to introduce an additional complication, and he was surprised that a legal objection on this point was not raised when the case came into

court. He understood, however, that if Mr. Bodmer were to receive a sample of preserved peas in their liquor, he would pour off the liquor as he did in the cases referred to. It seemed very necessary that some conclusion should be arrived at on this matter, and his own view was that, in the present state of the law, the only satisfactory way of stating the quantity of copper was to return the percentage of copper on the dry matter in the whole sample—peas plus liquor. It might be contended that the liquor was used in cooking, and this, indeed, was sometimes the case; and he thought it would not make a sufficiently strong case to base the analysis on the peas minus the liquor on the assumption that that was as nearly as possible the way in which they would be consumed, though no doubt this would be the most convenient way of dealing with the matter if it were legally possible to do so. He would be very glad if some conclusion could be come to on lines which would enable Mr. Bodmer's figures to be made use of.

He thoroughly agreed with Mr. Hehner in what he had said as to electrolytic methods. The method which he had himself found most satisfactory was the one followed by Mr. Bodmer, and he was bound to say that the colorimetric method with ferrocyanide was a very satisfactory way of doing the work. He was very much in favour of the ferrocyanide method of determining copper, both colorimetrically and gravimetrically.

Mr. HEHNER said that the main question turned upon what was actually sold. Was it the liquor plus the peas, or the peas alone? He had lately weighed a tin of peas marked "1 lb.," and found that the peas alone weighed 1 lb., the water being adventitious. If a butterman sold butter by the butter-fat, he could not well be proceeded against for adding water, and he (Mr. Hehner) thought that if it was the peas that were sold, and not the liquor, then it was correct to pour off the liquor and analyse the peas.

Mr. ALLEN said that in his opinion the certificate should be made applicable to the entire contents of the tin, but he would add to the results so stated the amount of copper which the peas would contain, exclusive of the water. This would afford additional information to the Bench, and show that the public analyst had taken the whole of the circumstances into consideration. He held the same opinion as Mr. Hehner with regard to the electrolytic method of determining copper. When incinerating such articles as peas for the determination of copper, it was desirable to char the substance moderately, and then add both sulphuric and nitric acid. This caused decomposition of any chlorides, and prevented possible loss of copper by volatilization. At the same time, the ash then consisted mainly of sulphates, and was far more manageable than where no sulphuric acid had been used. If the ash when obtained was moistened with a drop of sulphuric acid and nitric acid, and the product heated till fumes of sulphuric acid began to be evolved, the metals were left in such a state that on adding water any tin or lead was left entirely insoluble, while the zinc and copper dissolved.

Mr. CASSAL remarked that the fact that one grain of copper had been found in fresh peas appeared to him to be due to something abnormal. He had lately analysed several samples of French beans and peas imported into this country with a guarantee that they were not coloured with copper, without finding a trace of copper in any of

them, and it therefore seemed to him that such large quantities of copper pointed to the existence of special circumstances which were unexplained.

The PRESIDENT said that the Society was very much indebted to Mr. Bodmer and Mr. Moor for these results. It appeared to be highly desirable that more results should be collected, in order that Vedrödi's very remarkable figures might be criticised. For instance, 3½ parts in 10,000, or nearly 3 grains per lb., seemed an enormous quantity of copper to be found in wheat, though it was, of course, well known that vegetables grown in the neighbourhood of copper works, or on soils containing copper, would take up considerable quantities.

SOME ANALYSES OF WATER FROM AN OYSTER FISHERY.

By CHARLES E. CASSAL, F.I.C.

(*Read at the Meeting, February* 3, 1897.)

EARLY in 1895 I was asked to analyse certain samples of water which had been taken for the purposes of an investigation as to the alleged transmission of infectious disease by means of oysters. I was instructed to make a chemical examination of the samples as rapidly as possible, and to furnish only certain data. I therefore did not myself carry out any bacteriological tests, and these were undertaken by a distinguished bacteriologist, Dr. G. E. Cartwright Wood, whose results were subsequently published. Having regard to the erroneous views which still prevail in some quarters as to the present practical value of bacteriology in enquiries of this kind it may be useful to place the chemical facts on record, and to contrast the conclusions which could be arrived at by the aid of chemical methods on the one hand, and of bacteriological methods on the other.

We have heard a great deal about the failure of chemical analysis to afford correct and reliable information as to the pollution of water and the character of water-supplies, in certain cases; and it has become important to emphasize the facts that, at present at any rate, bacteriology, *per se*, can give us but little information of real value, and certainly cannot give us information of a decisive character.

Seven samples of water were submitted. They were taken in and about an estuary on the East Coast, where oysters are extensively cultivated, and the descriptions supplied with them were as follows:

"No. 1. P. Channel. Fattening bed for oysters. Bottom quite clean, no mud. Chance of contamination from the town of B. Example of an 'uncontaminated' oyster bed."

"No. 2. B. water. Taken opposite the town of B. A small amount of sewage runs into this water."

"No. 3. River C. a little above the estuary. Breeding ground for oysters. Nearest source of contamination is the town of W. above."

"No. 4. Taken in middle of channel opposite the town of W., and almost opposite the open end of the sewer of that town."

"No. 5. No description.

"Nos. 6 and 7 taken from oyster beds existing under the worst possible conditions."

The analytical results were as under:

	1.	2.	3.	4.	5.	6.	7.	
Date of collection	4.2.'95	4.2.'95	4.2.'95	4.2.'95	13.3.'95	13.3.'95	13.3.'95	
Total solid matters	3202·0	3330·8	3254·0	2106·4	3636·8	3642·0	3040·0	parts per 100,000
Chlorine as chlorides	1625·0	1737·5	1675·0	1025·0	1875·0	1887·5	1562·5	,, ,, ,,
=Chloride of sodium	2678·0	2863·0	2760·0	1689·0	3090·0	3110·0	2575·0	,, ,, ,,
Saline ammonia	0·148	0·040	0·130	0·568	0·084	0·090	0·460	parts per 1,000,000
Organic ammonia	0·136	0·218	0·180	0·286	0·214	0·108	0·140	,, ,, ,,
Oxygen absorbed from permanganate, 30° C., 4 hours	1·677	1·572	2·227	1·513	2·263	1·290	1·578	,, ,, ,,

If the results are carefully considered, with due regard to the descriptions of the places from which the samples were obtained, it is justifiable to conclude that in each case the data afford evidence of contamination; and, from a practical point of view, this is really all that is required in a case of this kind. It should be obvious that the water in which oysters or other shell-fish intended to be eaten raw are kept should be absolutely free from even the suspicion of pollution with sewage. My bacteriological colleague in the investigation has very properly pointed out that "the lesson to be learned from these and numerous similar observations is—that no molluscs should ever be permitted to be used as articles of diet which have been obtained from waters subjected to contamination, since this alone, apparently (without the presence of specific infective disease germs, such as those of typhoid fever or cholera), can give rise to most dangerous disturbances of the general health." He has further pointed out—and I am fully in agreement with him—that the only principle which can be applied in such an investigation is to judge the waters examined by the same standards as those which must be employed in judging drinking-water, so far as it is possible to do so.

The opinion given was to the effect that the results afforded evidence of contamination in each case, the data in the case of No. 4 showing pollution with matters partaking of the nature of recent sewage; and that, especially in view of the descriptions given with the samples, those numbered 2, 3, 6, and 7 yielded data indicating similar pollution to a lesser extent: while No. 1 could not be taken as affording an example of an uncontaminated water, the results in the case of this sample rather pointing to the conclusion that the oyster-bed near which it was taken was distinctly liable to "contamination from the town of B." For obvious reasons, the "oxygen absorbed" figures have not been regarded as affording evidence of much importance. They were determined and inserted in the report because they were asked for. I need not anticipate the usual, and very ancient, objections which may be raised against the drawing of the conclusions mentioned mainly from the ammonia figures; I have only to say that in this particular case these figures afford distinct and positive evidence of some contamination, which, in the circumstances named, may reasonably be regarded as due to "sewage" pollution.

I may now call attention to what could be done in the matter by the aid of bacteriology. Dr. Cartwright Wood informed me that the bacteriological examination had unfortunately afforded little or no guidance in the matter. In point of fact the

application of bacteriological methods *alone* yielded no infor
the condemnation or the passing of the samples would have bee
has stated that in judging of a water he would not trust to
alone, and that an adequate chemical analysis is necessary;
opinion, bacteriological examination frequently gives informat
great importance. A negative result from both chemical and b
tions does not, in Dr. Wood's view, in any case indicate freedo
hardly say that with these opinions I entirely concur. I do not
that I desire to condemn bacteriological processes as valueless.
contrary, that the results obtained by them, when applied
proper chemical analysis supplemented by microscopical examir
cases, be of considerable value. The application of chemical p
examination of water may be of doubtful value in certain ca
obtained, when considered alone, may mislead the investigator
over-confidence in bacteriological methods are far greater, and
ticularly threatening. I have nothing but condemnation for those who, upon
the results of inadequate analyses, habitually "pass" samples of w " perfectly
pure," "absolutely safe," and "excellent in quality." Such expressions are not
permissible under any circumstances; but what is to be said of bacteriologists who
"pass" waters upon negative bacteriological results alone?

Dr. Cartwright Wood has shown, by a series of very interesting experiments, that what may be called the "laboratory typhoid bacillus," and the "laboratory cholera bacillus" survive in sea-water, and that they can be recovered from oysters which have been purposely infected. Dr. Klein, according to a report to the Local Government Board, which forms part of the Report of the Medical Officer to the Board for 1894-95, appears to have proved the same thing. But while Dr. Cartwright Wood does not seem to attach any very great importance to the presence of the ubiquitous *Bacillus coli*, Dr. Klein attaches what I conceive to be an enormously exaggerated importance to it. The *Bacillus coli* is so universally distributed that the conclusions to be drawn from its presence are of very little value indeed; and, certainly, to draw absolute conclusions as to the existence of sewage pollution because a few of these bacilli have been found, is altogether unjustifiable, not to say absurd. No doubt the detection of a large number may afford confirmatory evidence, but that is all. The mere counting of colonies is, at the best, but a coarse and clumsy attempt to diagnose the existence of organic pollution, and by itself gives no further information than the coarsest and most clumsy of chemical processes. I contend again, as I have contended before both here and elsewhere, that no single instance can be adduced wherein the application of bacteriological methods has resulted in the detection of specific organisms, such as the typhoid bacillus or the cholera bacillus, in samples of water credited with having produced the one disease or the other. I do not mean to assert that improved bacteriological methods will not, in the future, enable bacteriologists to say with certainty that a water suspected of having produced typhoid or cholera contains, or does not contain, a germ which, when ingested by man, will, under favouring circumstances, produce either of these diseases; but I do say that up to the present time no evidence of any value has been adduced to show that this has been done. The

is essentially one which is liable to yield uncertain and mis-
l experience shows that a water yielding bacteriological results
ould justify its condemnation, will also yield results by chemical
ıpplemented by microscopic examination which would necessi-
nation. A point of considerable importance in regard to my
that, according to the report previously alluded to, Dr. Klein
ined a great number of oysters obtained from sources known to
ated with sewage, and that he found absolutely nothing of a
ept in one single case. On page 115 of the report he states that
which was described as a deep-sea oyster obtained from the
elded a pure culture of a motile cylindrical bacillus which he
ıtical with the typhoid bacillus.
he chemical results above recorded show, beyond possibility of
ers examined were contaminated, that this fact is sufficient for
ınd that bacteriological tests, carried out with the same samples,
e upon which condemnation would have been justified. The
a fact, afforded positive information; the bacteriological results did

NOTE.—Since the above paper was read, my friend, Mr. Leonard Archbutt, F.I.C., who was present at the meeting, has kindly sent me the results of two analyses of uncontaminated sea water which were made by him in 1884. Mr. Archbutt has kindly allowed me to introduce these results as an addendum to my paper. It will be seen that they fully confirm the view which I have put forward.

ANALYSES OF SEA WATER.

BY LEONARD ARCHBUTT, F.I.C.

Taken two miles off Bridlington Quay, between Bridlington and Flamborough, August 16, 1884.

	Pts. per million.
Free Ammonia	0·015
Albuminoid Ammonia	0 03

Taken off Robin Hood's Bay, near Whitby, four miles from land, August 30, 1884.

	Pts. per million.
Free Ammonia	0·005
Albuminoid Ammonia	0·03

Both samples were analysed in a fresh condition.

DISCUSSION.

Dr. STEVENSON said that on scanning the figures given it seemed to him that chemical analysis had thrown as little light upon these waters as bacteriological examination had. The estimation of sewage in sea-water was a very difficult matter, and a good deal more information was wanted in regard to it, in the direction of chemical analysis as well as of bacteriological examination.

Dr. J. A. VOELCKER said that he had been wondering when the time would come that there would be uniformity among chemists in the expression of their analytical results. It had taken him some time to calculate Mr. Cassal's figures into those that were familiar to him, and in the end he had come to much the same conclusion as Dr. Stevenson had.

There was one matter which Mr. Cassal had not mentioned, viz., the amount of nitrates in the samples. He did not know whether Mr. Cassal had the figures at hand as to the quantity of nitrates which these waters contained, but he thought they would be very material.

Mr. ALLEN desired to ask in what way Dr. Voelcker would like to see the results stated: He (Mr. Allen) thought that Mr. Cassal had hit upon a very happy way of putting the figures.

Dr. VOELCKER said that all he wished to see was uniformity of practice throughout. It seemed to him absurd to put down one factor in one notation and one in another notation; and equally absurd to adopt one system for sea-water and another for fresh water. A third system would probably be necessary in cases where sea-water and fresh water were mixed.

Dr. RIDEAL remarked that the figures of the No. 4 sample (which was evidently brackish water, containing more fresh water than any of the others) showed high saline and organic ammonia, while the oxygen absorbed was the lowest but one, and therefore the pollution, as measured by the oxygen absorbed, did not seem to throw much light on the relative value of the waters. It would have been interesting if Mr. Cassal could have given the saline and organic ammonia normally present in the sea-water of this district, free from any admixture of fresh water or possible pollution with land drainage.

With regard to the statement of results, his own practice was to give all data in parts per 100,000, which might not find acceptance in some quarters, but which, at all events, had the merit of uniformity and the approval of a British Association committee.

Mr. RICHMOND desired to ask whether, considering the large proportion of chlorides necessarily present in waters of this kind, Mr. Cassal put forward his figures given for oxygen absorbed as anything more than approximations.

The PRESIDENT said that the point referred to by Mr. Richmond was the well-known difficulty which was first pointed out before the Society by Dr. Dupré, in connection with the fact that when, as in the case of sea-water, there was a large proportion of chlorides present, chlorine was apt to be liberated by the permanganate, and this very greatly interfered with the accuracy of the results if the operation was carried out at the usual temperature of 80° F. Dr. Dupré had shown that in dealing with sea-water or brackish water it was necessary to work at a very low temperature and to use well-stoppered bottles.

In asking the meeting to thank Mr. Cassal for his paper, he would only say one word. He thought it a pity to too much disparage the help which bacteriology afforded, even if the present state of knowledge on this subject necessitated somewhat crude observations. He would certainly say for himself that waters were often submitted to him in which the chemical results were very doubtful, and in such cases the bacteriological results were of great assistance in the formation of an opinion. It was, at any rate, a known fact that in unpolluted deep well waters there was very little bacterial life, and the question of surface pollution finding its way into deep wells was one which often had to be dealt with. If only on this account, bacteriological examination must be regarded as a useful adjunct to

chemical analysis. Then, again, there was that class of waters the existence which was so prominently brought before the Society by Mr. Hehner and Dr. Dupr namely, surface wells highly contaminated in an exceedingly nitrifying soil, where there was very little free ammonia, scarcely any albuminoid ammonia, and scarcely any "oxygen absorbed." In such cases, as far as chemical results went, all there was to go upon was a proportion of chlorine which might or might not be significant (according to local circumstances), and the proportion of nitrates. In these cases the information afforded by the results of bacteriological examination was often of considerable value. Although no doubt the value of bacteriological methods was sometimes strained, it ought not to be under-estimated. He himself had subjected every water sample he had had for the past two years to a bacteriological examination, and he thought if everyone who had to analyse water would do this (without, however, in the present state of the science, attempting to look for a typhoid or following any other practically wild-goose chase of that kind), they would find the practice a very useful one.

It seemed to him a pity that there should be so many divergencies in the statement of the results of water analyses. The old fashion of stating both grains per gallon and parts per million had crept into use when Mr. Wanklyn wrote his little book on "Water Analysis." Mr. Wanklyn stated the total solids in grains per gallon, and also the chlorine, and the ammonia in parts per million, to make the figure look a little more tangible perhaps. Our Society's Water Committee had recommended, a good many years ago, a scale which was still followed, he might almost say, by every member of the Society excepting Mr. Cassal. According to that scale, every constituent was stated in grains per gallon. Dr. Frankland had introduced parts per 100,000, which was still used by some of his old pupils, but he (the speaker) thought that the majority of analysts stated their results in grains per gallon.

Mr. CASSAL said it would be seen that the sample No. 6, which was the most sea-watery of all the samples, was the cleanest with respect to organic contamination, and taking this as a sample for comparison, it was perfectly obvious that there was some organic contamination in the other samples.

With regard to Mr. Richmond's question, he did not attach great importance to the oxygen absorbed figure. He made the determinations rather as a matter of routine, and because they were asked for, and, having the results, had put them down, but it was clear that they did not afford as much information as in other cases.

The object to be kept in view in stating analytical results was to give *effective* figures—figures which were effective for the formation of an opinion. As to giving results in grains per gallon, he was sorry to find himself obliged to argue against the use of such an utterly unscientific method. "Grains" and "gallons" were barbarous measures which men of science ought to avoid the use of, whenever possible. The recommendation of the Water Committee on this point ought never to have been made. He was sorry that he could not agree with Drs. Stevenson and Voelcker, and he hoped that those gentlemen would alter their opinions on further consideration.

The statement of results was a subject that had often been discussed before the Society. He had endeavoured in the case of these samples to give as clear an idea as possible of them, and at the same time to avoid stating ineffective figures.

He did not wish the President to think that he disparaged bacteriological results. He was merely anxious to prevent misconception in regard to their value, and to prevent, if possible, reliance being placed upon them when they were put forward without any other results.

NOTES ON ALCOHOL.

By J. F. Liverseege.

(*Read at the Meeting, April 7, 1897.*)

I. Correction of Specific Gravity for Expansion.

In taking the specific gravity of more or less dilute solutions of alcohol, it is often convenient to adjust the volume of the liquid at a temperature a few degrees from 60° F., and then correct the result to 60° F. In Allen's "Commercial Organic Analysis" the following formula is given :

$$\text{Correction of specific gravity for } 1^\circ \text{ C.} = 0{\cdot}00014 + \frac{1 - \text{sp. gr.}}{150}.$$

In Hoffmann and Power's "Chemical Analysis" the correction for 1° C. is stated to be equal to 0·4 per cent. of absolute alcohol. I have calculated from these rules the correction for alcohol of several specific gravities for 1° C., and give for comparison figures taken from the table in Thorpe's "Dictionary of Applied Chemistry" (i., 45), and from that by Lyons (*Phar. Jour.* [3], xvi., 824) :

Specific gravity ...	0·85	0·88	0·92	0·95	0·98
Allen	0·00114	0·00094	0·00067	0·00047	0·00027
Hoffmann and Power	0·0010	0·0009	0·0008	0·0008	0·0005
Thorpe	0·00084	0·00083	0·00078	0·00065	0·00021
Lyons	0·00083	0·00081	0·00075	0·00061	0·00025

Owing to the differences in these results, and the amount of interpolation required in the tables of Thorpe and Lyons, an attempt was made to construct a detailed table from published work on the expansion of alcohol of various strengths. In the classical tables of Gilpin (*Trans. Royal Soc.*, 1792, 425), the weight of alcohol of various strengths contained in a 2965 grain specific gravity bottle is given for every 5° F. from 35° to 100° F. ; from the results for the temperatures from 50° to 70° F. the correction for 1° F. was obtained for forty strengths of alcohol. Another set of results was obtained from Mendeleef's specific gravity tables for 10° and 20° C., as given in Watts' "Dictionary of Chemistry." The figures of Squibb (*Phar. Jour.* [3], xv., 46, from "Ephemeris") and Tralles (Table III.) were compared with these, and also with the tables of Thorpe and Lyons before mentioned.

The result of these comparisons showed that when more than 80 per cent. of alcohol was present the corrections given by the various authorities sometimes differed by more than 0·00001 for 1° F., but that for spirits containing less alcohol than this the agreement was remarkably close. The following table is compiled from these results ; multiplication of the figures given by 1·8 will give the correction for 1° C. :

CORRECTION OF SPECIFIC GRAVITY OF ALCOHOL FOR TEMPERATURE.

Specific Gravity.	1° F. =	Specific Gravity.	1° F. =
0·794—0·864	0·00046	0·965—0·966	0·00026
0·864—0·889	45	0·966—0·967	25
0·889—0·902	44	0·967—0·968	24
0·902—0·912	43	0·968—0·969	23
0·912—0·921	42	0·969—0·970	22
0·921—0·928	41	0·970—0·971	21
0·928—0·935	40	0·971—0·973	20
0·935—0·940	39	0·973—0·974	19
0·940—0·943	38	0·974—0·975	18
0·943—0·946	37	0·975—0·976	17
0·946—0·949	36	0·976—0·977	16
0·949—0·951	35	0 977—0·978	15
0·951—0·953	34	0·978—0·980	14
0·953—0·955	33	0·980—0·981	13
0·955—0·957	32	0·981—0·983	12
0·957—0·959	31	0·983—0·985	11
0·959—0·961	30	0·985—0·987	10
0·961—0·962	29	0·987—0·990	0·00009
0·962—0·963	28	0 990—0·995	8
0·963—0·965	27	0·995—1·000	7

When the temperature is lower than 60° F. the correction given, multiplied by the difference in temperature, is to be subtracted from the specific gravity found; but when the temperature is higher, added to it.

II. Different Methods of stating Alcoholic Strengths.

The alcoholic strength of liquids is generally expressed as the percentage by weight or by volume of absolute alcohol present, or as the percentage of proof spirit by volume. Below are given formulæ for the calculation of each statement to any of the others, as well as to grammes of alcohol per 100 c.c., which is sometimes required. The formulæ are based on Squibb's absolute alcohol of specific gravity 0·7935, proof spirit containing 49·2 per cent. of this alcohol and having a specific gravity of 0·9198.

S = specific gravity at 60°/60° F.
% = grammes of absolute alcohol per 100 grammes.
v/v = c.c. absolute alcohol per 100 c.c.
w/v = grammes of absolute alcohol per 100 c.c.
P = c.c. proof spirit per 100 c.c.

(C.c. is here used to indicate the volume of 1 gramme of water at 60° F.)

$$\% = \frac{v/v \times 0{\cdot}7935}{S} = \frac{w/v}{S} = \frac{P \times 0{\cdot}4525}{S}$$

$$v/v = \% \times 1{\cdot}262\ S = 1{\cdot}262\ w/v = 0{\cdot}5703\ P$$

$$w/v = \% \times S = 0{\cdot}7935\ v/v = 0{\cdot}4525\ P$$

$$P = \% \times 2{\cdot}21\ S = 1{\cdot}753\ v/v = 2{\cdot}21\ w/v$$

Grains per fluid ounce = w/v × 4·3756, as one fluid ounce contains 437·5 grains of water at 62° F.

Statements of three important strengths of spirit are here given as calculated by these formulæ:

Absolute alcohol,	specific gravity	0·7935 = 100 % = 100 v/v	= 79·35 w/v = 175·4 P
Rectified spirit	,, ,,	0·838 = 84 % = 88·84 v/v	= 70·39 w/v = 155·6 P
Proof spirit	,, ,,	0·9198 = 49·2% = 57·03 v/v	= 45·25 w/v = 100·0 P

III. Calculation of the Results of Distillation.

If, in the distillation of an alcoholic liquid of specific gravity S L c.c. and G grammes are taken, and the distillate measures L′ c.c. and weighs G′ grammes, and has a specific gravity S′, and contains B per cent. by weight, or D per cent. by volume of absolute alcohol, or F per cent. by volume of proof spirit, as shown by an alcohol table, then the quantity of alcohol present in the liquid can be calculated by the following formulæ:

$$\% = \frac{B\,L'\,S'}{L\,S} = \frac{B\,G'}{G} = \frac{B\,G'}{L\,S}$$

$$v/v = \frac{D\,L'}{L} = \frac{D\,G'\,S}{G\,S'} = \frac{D\,G'}{L\,S'}$$

$$w/v = \frac{B\,L'\,S'}{L} = \frac{B\,G'\,S}{G} = \frac{B\,G'}{L}$$

$$P = \frac{F\,L'}{L} = \frac{F\,G'\,S}{G\,S'} = \frac{F\,G'}{L\,S'}$$

The first formula in each series is to be used when both liquid and distillate are measured, the second when they are both weighed, and the third when the liquid is measured and the distillate weighed. The above formulæ assume that the liquid and distillate are at 60° F. when measured, but for the percentage of proof spirit the correction for expansion may be made as follows, where t is the temperature in degrees Fahrenheit, and C the apparent co-efficient of expansion of the liquid, and C′ that of the distillate for 1° F. The co-efficient of expansion is equal to the correction of the specific gravity, as given in the foregoing table, divided by the specific gravity.

$$P = \frac{F\,L'[1+(60-t')C']}{L[1+(60-t)C]}.$$

If the liquid is at t° F., and is assumed to be at 60° F.

$$\text{True result} = \text{calculated} - \frac{(60-t)F\,L'\,C}{L+(60-t)C\,L'}.$$

If the distillate is at t'° F., and is assumed to be at 60° F.

$$\text{True result} = \text{calculated} + \frac{(60-t')F\,L'\,C'}{L}.$$

Applying these formulæ to cases of distillation will show that, if 50 c.c. of proof spirit are diluted and distilled to 100 c.c., an error of 1° F. in measuring the spirit will show 0·05° O.P., and that the same error in temperature in measuring the distillate will give 0·03° O.P. instead of proof strength. If 25 c.c. of whisky are diluted and distilled to 25 c.c., the result of an error of 1° F. will give 75·03 per cent. instead of 75 per cent. of proof spirit.

Corrections may also be made for errors in the calibration of the measuring vessels. If the pipette delivers L + l c.c., and is assumed to deliver L c.c. of liquid, true result = calculated $- \frac{F\,L'l}{L(L+l)}$.

If the specific gravity bottle holds L′ + l′ c.c, and is assumed to contain L′ c.c. of distillate, true result = calculated $+ \frac{F\,l'}{L'}$.

If in the measurement of proof spirit mentioned above there is an error of 0·01 c.c. in 50 c.c., the proof spirit will appear to be 0·02° O.P., or 0·01° O.P. if that error is made on the 100 c.c. of distillate. If the same error is present in the 25 c.c. specific gravity bottle, the whisky will appear to contain 75·03 per cent. of proof spirit. In most estimations errors of this magnitude are negligable. In each case it is assumed that the correct specific gravity is used for comparison with the alcohol table.

IV. Indirect Estimation of Alcohol.

Calculation of the amount of alcohol present, from the specific gravity of a liquid and from the quantity of solid matter it contains, may be used to check the results of distillation, and is particularly useful when the presence of volatile oil renders distillation unsatisfactory.

If 100 c.c. of a liquid of specific gravity S is composed of E grammes, or E′ c.c. of solid matter, and A grammes, or A′ c.c. of alcohol and water, and the specific gravity of the dilute spirit is S″, and if the ratio $\frac{S - S''}{E} = K$, where K is a constant for all usual strengths of that liquid, then $S = \frac{E + A}{E' + A'}$; and as $A' = 100 - E'$, and $A = S''(100 - E')$, and also $S - S'' = EK$, these values may be substituted, giving

$$E' = \frac{E(1 - 100K)}{S''}.$$

Tincture of iodine may be taken as an example of the application of these formulæ. I have shown ("Year-Book of Pharmacy," 1894, 477) that K = 0·008. A tincture contained 4·64 w/v of iodine and iodide of potassium, and had a specific gravity of 0·878.

The specific gravity of the contained spirit (S″) = 0·878 − 4·64 × 0·008 = 0·841.
The volume occupied by the solids (E′) = $\frac{4{\cdot}64(1 - 100 \times 0{\cdot}008)}{0{\cdot}841} = 1{\cdot}2$ c.c.

As specific gravity 0·841 = 82·9 per cent. of absolute alcohol, the tincture contained 82·9 × 0·841 × 98·8 = 68·9 w/v of absolute alcohol, while 68·6 w/v was actually present.

The volatile oil present in tincture of myrrh makes the estimation of the alcohol by distillation very unsatisfactory. I found that even after dilution and precipitation with calcium chloride and sodium phosphate, the distillate contained enough volatile oil to make the result decidedly too low. Seven samples of myrrh were made into eleven tinctures with spirit of known strength, and the solid matter and specific gravity of the finished tincture determined. From these results values of K were obtained. Nine of them varied from 0·0028 to 0·0033, one was 0·0024, and one

0·0034; as a mean result 0·003 was taken. Experiments were also made (*v. Chemist and Druggist*, xlviii., 558) which showed that the effect of the small amount of water contained in the myrrh, and of the difference of the solid matter in solution and the extract as weighed, did not seriously affect the result.

For whisky K has been found to equal 0·0055. For example, S was found to be 0·9541, and E to be 0·26 w/v. $S'' = 0{\cdot}9541 - 0{\cdot}0055 \times 0{\cdot}26 = 0{\cdot}9527$, while 0·9530 was found by distillation. This method has been found useful for checking the results of distillation; the difference between the two rarely exceeds 0·0005, and is generally less than this. As whisky rarely contains more than 0·5 per cent. of solid matter, its volume may be neglected, and the proof spirit corresponding to S″ may be taken as the alcoholic contents of the whisky.

ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

FOOD AND DRUGS ANALYSIS.

The Determination of Albuminoids in Milk. A. Schlossmann. (*Zeit. Physiol.*, xxii., 197; through *Zeit. ang. Chem.*, 1897, 128.)—Ten c.c. of the milk are warmed to 40° C. with 3 to 5 parts of water, and 1 c.c. of a concentrated solution of alum added. Should the flocculent precipitate not subside rapidly, an additional 0·5 c.c. of the alum solution is used, since a slight excess (up to 1 c.c.) does not affect the result. The precipitate (which in the case of cow's, goat's, pig's, and ass's milk is in medium-sized or large flakes, but in woman's milk in small flakes) is allowed to stand for some minutes, and then filtered. After having been well washed with water, the filter and its contents are extracted with ether in a Soxhlet apparatus, the nitrogen determined by Kjeldahl's method, and the amount calculated into casein.

To the clear filtrate from the casein 10 c.c. of tannin solution are added, the voluminous precipitate filtered off, washed three times with water, and the nitrogen it contains determined, and expressed as that of the soluble albuminoids (albumin and globulin).

The separation of the casein in human milk may be accelerated by the addition of a little sodium chloride during the warming, and the filtration by the addition of calcium phosphate, which mechanically prevents the fine casein flakes from passing through the filter. C. A. M.

On Mixing Dimethylamidoazobenzene with Margarine for the Purpose of affording Evidence of Identity. A. Partheil. (*Chem. Zeit.*, xxi., No. 27, 255.)—A proposal was made ten years ago by Soxhlet to make it compulsory upon margarine manufacturers to mix 1 gramme of phenolphthalein with every 100 kilos of margarine, in order to impart a "latent" colour to the product, rendering it easy of identification. There are, however, certain practical drawbacks to this plan. The phenolphthalein can easily be extracted; there is the liability to discoloration when the margarine is packed in wooden tubs which have been washed with soda; etc.

The author proposes dimethylamidoazobenzene in place of phenolphthalein. Margarine mixed with dimethylamidoazobenzene acquires a rose colour on adding a little dilute sulphuric or other inorganic acid. The proposal was brought before the chairman of the German Imperial Commission on Margarine in February, 1896, who submitted it to the Department of Public Health, with the result that the Government appeared to be favourably disposed towards the proposal.

Experiments have been made upon dogs by Professor Munk, of Berlin, who reports that long-continued dosing produced no harmful effects, and that even the administration of very large quantities produced only the same effects as corresponding doses of common salt or sugar.

Should the proposal be adopted, it will be possible for every housewife to distinguish margarine from butter, and even to detect the adulteration of butter with margarine, in very flagrant cases. H. H. B. S.

The Analyses of Butter by the Determination of the Critical Temperature. L. Crismer. (*Bull. de l'Assoc. belge des Chim.*, 1897, x., 453-481.)—In order to fix the limits of this constant, the author has made full analyses of 173 samples of butter during the course of 1896. The mean critical temperature for the twelve months was 52·5°, and the acidity of 2 c.c. of the fat was equal to 3 c.c. of $\frac{N}{20}$ alkali, making the critical temperature of the neutralized butter 55·5° with absolute alcohol. For the last six months of 1895, the corresponding figures were: Critical temperature, 52°; acidity, 2·6; critical temperature of neutralized butter, 54·6° As was pointed out in a previous communication (ANALYST, this vol., 72), the critical temperature of the neutralized butter is that of the acid butter plus the number of c.c.'s of $\frac{N}{20}$ alcoholic potash required to neutralize 2 c.c. of the butter-fat. The critical temperature was found to increase in August, it continued to rise throughout September, and reached its maximum in October.

It was shown before (ANALYST, xx., 257) that there appeared to be a certain relation between the Hehner number and critical temperature. This relation has now been confirmed, and is expressed in the following table:

		Alcohol with 9 per cent. Water.		Absolute Alcohol.	Insoluble Fatty Acids.
Critical temperature,	below ...	100°	or	54°	86—88
,, ,,	from ...	100°—108°	,,	54°—62°	88—90·5
,, ,,	,, ...	108°—118°	,,	62°—72°	90—93·3
,, ,,	,, ...	118°—124°	,,	72°—78°	93—95·5

Similarly, a ratio has been established between the Reichert-Meissl number and the critical temperature. The limits of the former for pure butter are 27-28 and 20-21, whilst the value for margarine is 3-4. The difference between the higher limit for butter and margarine = 24. With alcohol containing 9 per cent. of water, the difference between the critical temperatures of margarine and butter is 124 – 100 = 24; and with absolute alcohol, 78 – 54 = 24. Hence the sum of the Reichert-Meissl value and critical temperature of the neutralized butter gives a constant number. A long table is given, showing that with alcohol of specific gravity 0·8195 this constant is 129, and with absolute alcohol 83·5. Therefore the Reichert-Meissl value may be

obtained by deducting the critical temperature from the constant 129 or 83·5, according to the strength of alcohol employed. For example, a butter showed a critical temperature of 55° in an open tube (see ANALYST, this vol., 71) with absolute alcohol. The acidity was 2 c.c., making the critical temperature of the neutralized butter 57°. Hence the Reichert-Meissl value was 83·5° – 57 = 26·5, which result was confirmed by distillation.

With regard to the examination of butter with the refractometer, the author gives a number of results to prove that it may be misleading, and that adulterated butters may give lower figures than genuine samples. The following examples, in which the readings were taken at 40° C., are taken from his table:

	Abbe-Zeiss refracto-meter.	Insoluble Fatty Acids.	Critical Temp. + Acidity.
Butter adulterated	45·2	91·48	66·3°
,, ,, ...	45·6	91·48	67·5°
,, pure	45·8	—	57°
,, ,, ...	46	—	59·5°

The general conclusion arrived at is that, whilst this is a rapid and accurate method of arriving at the ordinary analytical data, the variation of the constant in different specimens of genuine butter is too great to allow of any certainty in the detection of small additions of margarine.

See also ANALYST, xx., 209; xxi., 241; and xxii., 21. C. A. M.

Quick Method for detecting Margarine in Cheese. R. Hefelmann. (*Zeit. für offent. Chemie*, iii., 117.)—Twenty to 50 grammes of the cheese are either grated or rubbed down with a little sand, according as it may be hard or soft, and placed in a test-tube with 20 to 25 c.c. hydrochloric acid of specific gravity 1·19. The tube is then immersed in a bath of boiling water, and heated for about half an hour, with repeated shaking. The casein dissolves to a brown or violet-red fluid, while the butter-fat separates as a clear layer, floating upon the acid solution. A drop of the fat is then removed by means of a dropping-tube, and examined by the butter refractometer of Wollny. Should the proportion of fat be too small to separate in this way, the contents of the tube may be cooled to 30° C., and 15 c.c. of petroleum ether (boiling below 70° C.) added, the whole shaken, and the petroleum ether with the fat in solution transferred to a beaker and evaporated, and the residual fat examined by the refractometer.

On continued heating, the refraction of the lower fatty acids gradually increases, until in some cases it ultimately reaches the lowest observed value for margarine. Further, in certain cases the fat of poor cheeses may show refractometer numbers up to 2·5 above the doubtful point. H. H. B. S.

Black Discoloration of a Cheese. C. Besana. (*Chem. Zeit.*, xxi., No. 28, 265.)—A sample of Parmesan cheese, covered with deep black marks resembling ink-stains, and possessing a smell like garlic, was sent to the author for investigation. A microscopical examination revealed nothing, but chemical analysis showed that the

stains were due to ferrous sulphide. The author came to the conclusion that the odour was caused by sulphur compounds, resulting from the decomposition of the protein substances, and that the iron contamination had its origin either in the water used for washing the vessels, in the vessels themselves, or in the saffron or rennet used. H. H. B. S.

The Estimation of Starch and Glycogen in Meat Preparations. A. Bujard. (*Forsch. Ber.*, 1897, iv., 47, 48.)—For the determination of glycogen, the author prefers Mayrhofer's method (*Forsch. Ber.*, 1896, 141), which he considers more simple than that of Niebel-Salkowsky (*Zeit. Fleisch und Milchhyg.*, 1891, 185). The flesh to be examined is dissolved in aqueous potash, albuminoid matters precipitated by means of hydrochloric acid and Nessler's reagent, and the glycogen, after precipitation by the addition of alcohol to the clear filtrate, is washed on a weighed filter with dilute alcohol and ether, and dried at 110° C.

The results given in I. are quite recent, whilst those in II. were obtained some time ago by the Niebel-Salkowsky method.

I.

	Water per cent.	Per cent. Glycogen Direct.		Per cent. Glycogen calculated on Dried Substance.	
		Niebel.	Mayrhofer.	Niebel.	Mayrhofer.
Horseflesh	74·44	0·440	0·445		
	74·87	0·600	0·520		
	76·17	1·827	1·727		
	76·00	0·592	0·610		
Red Sausage (Knackwurst)	69·26	—	0·038*		
Pork Sausage	67·25	—	0·24*		
Veal ...	74·6	—	0·086		
Pork ...	75·0	—	0·186		

II.

	Per cent.				Per cent.				Per cent.		
	Water.	Glycogen Direct.	Glycogen on Dried Substance.		Water.	Glycogen Direct.	Glycogen on Dried Substance.		Water.	Glycogen Direct.	Glycogen on Dried Substance.
Horseflesh	61·83	0·846	2·24	Beef	75·55	0·018	0·073	*Horse Sausages:*			
,,	72·90	0·174	0·64	Veal	76·12	0·346	1·44	Red Sausage ...	70·04	0·504	1·68
,,	70·47	1·366	4·62	,,	74·47	0·066	0·25	Liver Sausage	67·00	1·762	5·34
,,	71·84	0·59	2·09	Pork	54·05	trace	trace	Salami ...	33·60	0·034	0·05
Horseflesh smoked	43·00	0·108	0·19	,,	66·29	—	—	*Sausages:*			
Beef (ox)	73·62	0·206	0·74					Salami ...	20·00	trace	trace
								Thuringian ...	12·93	,,	,,
								,, ...	29·16	,,	,,

* In these pepper-starch could be detected microscopically, and on testing with iodine only the blue starch reaction could be obtained, whilst in all the other cases the glycogen reaction was marked.

In the author's opinion, these figures show that only in exceptional cases (where the amount is large) can the glycogen be taken as conclusive of the presence of horse-flesh, especially when the latter is mixed with other kinds of flesh. C. A. M.

Examination of Chloroform. A. Béhal and M. Francois. (*Jour. Pharm. Chim.*, 1897, v., 417-424.)—Chloroform is readily obtained pure, but rapidly alters under the influence of light and air, producing among other substances the gas carbon oxychloride. In order to prevent this decomposition, manufacturers are in the habit of adding a small amount of alcohol as a preservative. It is this alcohol, with more or less water, which constitutes the principal impurity in chloroform, and which interferes with the tests prescribed by the French Codex. The authors have found the following method of analysis satisfactory:

Detection of Water.—All the samples of chloroform examined, when submitted to a temperature of – 20° to – 40° C. by the authors, deposited crystals, which on examination proved to be ice. The simplest test is to bring some of the crystals into contact with mercuric ammonium iodide, when the latter is decomposed by the water and produces red mercuric iodide. Chloroform freed from water does not give any crystals when exposed to the low temperature, but the addition of 5 parts of 98 per cent. alcohol in 1,000 causes them to reappear.

Estimation of the Alcohol.—A modification of Nicloux's method (*Bull. de la Soc. Therap.*, iii., 860) is employed. Ten c.c. of chloroform are shaken in a separatory funnel with 4 c.c. of sulphuric acid, which combines with the alcohol. The shaking out is again repeated with 4 c.c. of sulphuric acid and then with 2 c.c. The sulphuric acid solution is mixed with 40 c.c. of water and gently distilled until the distillate amounts to 20 c.c. By this means the ethyl sulphuric acid is decomposed into sulphuric acid and alcohol.

Five c.c. of the distillate are boiled with 2 c.c. of sulphuric acid and some water, and bichromate solution (16·97 grammes per litre) is added from a burette drop by drop until a green, but not greenish-yellow, tint is reached. Each 2 c.c. of the solution corresponds to 0·01 gramme of absolute alcohol. This process is accurate if the strength of the alcoholic solution does not exceed 2 c.c. per 1,000; but if the result obtained indicates a higher amount than this, it is necessary to dilute the distillate with distilled water, and to repeat the determination.

The test experiments with pure chloroform to which a known volume of absolute alcohol had been added gave concordant and accurate results. In eight different samples of chloroform intended for anæsthetic purposes the authors found from 3 to 12·5 c.c. per litre of absolute alcohol, the mean being 6·2 c.c.

When freed from alcohol as above, the samples of chloroform answered all the requirements of the Codex. Chloroform free from alcohol ought to distil completely at 60·8° C., and the distilled chloroform should have a density of 1·500 at 15° C. In the authors' opinion, the addition of a small amount of alcohol as a preservative should be permitted. C. A. M.

Rapid Method of Analysing Guiacol and Creosotes. L. Adrian. (*Bull. de Thérap.*, January 8, 1897; through *Journ. Pharm. Chim.*, 1897, 174-176.)—This method is based on the colour reaction which nitrous acid gives with pure guiacol. From 5 to 6 grammes of the drug are shaken in a 200 c.c. flask with water (exact quantity immaterial) for two minutes, and the liquid filtered. The filtrate is diluted with water, and to 1 c.c. are added 2 drops of a solution of sodium nitrite (10 per cent.) and 1 drop of nitric acid. Pure guiacol gives an orange colour, whilst samples containing less than 50 per cent. give a quite distinct yellow colour. By preparing standard mixtures of pure guiacol and creosote, and comparing the colours given by these with that yielded by the sample, it is possible in a few minutes to judge of the approximate purity of the drug. C. A. M.

TOXICOLOGICAL ANALYSIS.

Post-Mortem Examinations for Atropine. P. Soltsien. (*Zeit. für öffent. Chemie*, iii., 115.)—Dragendorff's directions were followed in the detection of atropine in the organs and urine of a child. The importance of examining the urine in such cases is shown, and attention is directed to the extreme sensitiveness of the physiological test on the eye for identifying the extracted alkaloid. Dilatation of the pupil was obtained where the Vitali test failed to give any indication of atropine.

H. H. B. S.

ORGANIC ANALYSIS.

Determination of Glycerin in Crude Glycerin and Spent Lyes. F. Filsinger. (*Zeit. für öffent. Chemie*, iii. 97.)—While formerly the trade in glycerin was confined exclusively to the product obtained in the manufacture of stearic acid candles, etc., a large trade is now carried on in the spent lyes of soap works, from which the glycerin used in the manufacture of explosives is distilled. The determination of pure glycerin in stearin glycerin is only occasionally required, the specific gravity, boiling-point and ash, taken in conjunction with a qualitative examination, being usually sufficient. In the examination of lyes, however, this procedure does not suffice, and recourse must be had to the determination of the pure glycerin present.

The first method to come into use for technical purposes was Flemming's, which is still used by a few pharmacists. The sample is neutralized with hydrochloric acid, boiled with a slight excess of lime, filtered and concentrated to the consistence of a syrup. The glycerin is then dissolved by a mixture of absolute alcohol and ether, the solution filtered, washed with alcohol and ether, evaporated and dried at 100° C., the weighed residue, less the ash (separately determined), giving the glycerin. The weak points of the process are, first, that certain foreign organic bodies dissolve in ether-alcohol, and, secondly, that a loss is sustained in evaporation. The question, however, has been fully discussed by Hehner.

Benedict and Zsigmondy's permanganate process, founded upon the earlier

proposal of Fox and Wanklyn, is now largely used, but the process as originally devised requires modification. Instead of the calcium oxalate being converted into carbonate and then determined volumetrically, the oxalate should be dissolved in dilute sulphuric acid and titrated with permanganate. The following are working directions for determining the amount of glycerin in spent lyes by this process: 5 to 20 grammes of the sample, according to the proportion of glycerin present, are diluted, acidified with hydrochloric acid, boiled, cooled, filtered, and made up to 250 c.c. A portion corresponding to 0·2 to 0·4 gramme glycerin is transferred to a flask with about 200 c.c. water and 10 to 12 grammes potassium hydrate. Finely-powdered potassium permanganate is now added, a little at a time, until the permanent change of colour takes place. The flask is then placed on a water-bath for ten to fifteen minutes, which causes the manganese precipitate to settle out, leaving the solution red. The solution is now decolorized by the cautious addition of a solution of sodium sulphite, the solution filtered, the precipitate washed with hot water, the filtrate acidified with acetic acid and evaporated to about 100 c.c. The solution is washed into a beaker, the oxalic acid precipitated by solution of calcium acetate, the precipitate allowed to settle, filtered off, washed, dissolved in warm dilute sulphuric acid, and the oxalic acid determined by titration with potassium permanganate.

Benedikt and Cantor's acetin method, published in 1888, was used in Germany for a long time side by side with other processes, but has now been given up, owing to its unsuitability in certain cases. A method, which the author made the subject of a paper in the *Chemiker Zeitung* in 1890 (xiv., No. 102) has been used for some years by the Dynamite Company of Hamburg, and is carried out as follows: 20 grammes are dried at 100° C. for eight to ten hours, cooled and weighed. To ensure complete drying, it should again be heated for some hours and re-weighed, though, as a rule, the loss suffered the second time amounts only to a few centigrammes. The loss in weight represents the water present in the sample. For the determination of the other impurities, 5 grammes are slowly evaporated at 180° to 190° C. in a platinum basin, and the residue cooled and weighed; but in this case also it is advisable to heat a second time and re-weigh, to make sure that the weight remains constant. The water and the residue deducted from the crude glycerin gives the pure glycerin by difference. Though open to the objections to which all indirect processes are subject, the method is free from serious sources of error, and has been found in practice to give satisfactory results.

The author mentions in conclusion the proposals of Planchon, Legler, and Hehner, in which the weight of the carbonic dioxide yielded on oxidation is made to indicate the percentage of glycerin. For the successful working of all three methods, however, the glycerin must be free from foreign organic bodies, and in the case of Hehner's method the removal of chlorides is necessary. H. H. B. S.

New Method for the Determination of Glycerin. F. Bordas and S. de Rackowski. (*Ann. de Chimie Analyt.*, ii. [2], 26-28.)—This is a modification of the method for estimating glycerin proposed by Legler (ANALYST, xii., 14), and modified by Hehner (*ibid.*, xii., 44), in which the authors utilize the transition of

the tint of the solution from yellow-green to blue-green for ascertaining the end-point of the reaction. They employ a potassium dichromate solution containing 48 grammes per litre, 1 c.c. of which corresponds to 0·005 of glycerin in 5 c.c., or 1 part per 1,000. The end-point is very easily determined between the limits of 0·1 and 2 grammes of glycerin per litre, 0·1 c.c. of liquid being sufficient to effect the colour change; but outside these limits the solution requires concentration or dilution, the end-reaction being less delicate. C. S.

Estimation of Gelatin. F. Jean. *Ann. de Chimie, Analyt.*, ii. [2], 25.)—One gramme of gelatin is steeped in cold water and dissolved on the water-bath, and made up to a 100 c.c. solution; one-tenth of this is mixed with 10 c.c. of a 1 per cent. solution of pure tannin, and the whole agitated with 5 grammes of sodium chloride and 1 gramme of acid sodium carbonate, to render the gelatin tannate insoluble. After passing through a quick-running filter, the liquid is collected in a glass graduated at 45 and 60 c.c., the first-named volume being made up by washing the precipitate with a solution of sodium chloride, having a specific gravity of 1·184. A solution of iodine containing 4 grammes per litre is then added, drop by drop, until the starch test reveals the presence of free iodine, whereupon the liquid is made up to the larger volume with distilled water, and more iodine solution added until a faint blue coloration is imparted to starch. In this manner the excess of tannin is titrated, and by deduction from the original quantity gives the amount required to precipitate the gelatin. The tannin solution should always be standardized, as tannin varies in purity. C. S.

Separation of Organic Bases by Means of Potassium Bismuth Iodide. E. Jahns. (*Chem. Zeit.*, xxi., No. 27; *Repertorium*, 75.)—The author's experiments show that potassium bismuth iodide is an excellent precipitant for bases soluble in water, these can be separated almost quantitatively from extracts by its use. The reagent, which was prepared according to Kraut's directions (*Lieb. Ann. Chem.*, 1882, 210, 310), revealed the presence of choline in a number of medicinal plants, in which it had not hitherto been found; thus, choline was found in chamomile flowers, milfoil (*Achillea*), leaves of the mallow (*Malva*), anise-seed, and bark of the elder. The so-called new alkaloid "bursine," found by Bombellin (*Pharm. Zeit.*, 1888, xxxiii., 53) in *Capsella* (shepherd's purse), proved to be choline. Choline was also found in moderately large quantities in the seeds of *Lathyrus sativus* and *Lathyrus cicera*. H. H. B. S.

Determination of Iron in Blood. A. Jolles. (*Chem. Zeit.*, xxi., No. 23; *Repertorium*, p. 60.)—A weighed quantity of blood is dried in a porcelain basin and heated gently over a Bunsen burner. As soon as the mass begins to swell, the basin is cooled, and nitric acid added drop by drop. An energetic reaction sets in, and the basin must be covered to avoid loss by spurting. When the action has subsided, the basin is placed upon a triangle and heated, at first gently, but by degrees more strongly, by means of the Bunsen burner. After heating for about half

an hour over the full flame of the burner, the basin is allowed to cool, a few more drops nitric acid added, and heat again applied, gradually, as before. This operation is repeated several times, until the contents of the basin are completely incinerated. Hydrogen potassium sulphate is then added in the proportion of 1 gramme to every c.c. of blood taken, and the mixture fused. After cooling, the contents of the crucible are washed out with hot water, acidified with dilute sulphuric acid, zinc (of known iron content) added, and the iron determined by titration with $\frac{N}{50}$ or $\frac{N}{100}$ permanganate in the usual way.

The author further describes a simple method for determining iron in blood for clinical purposes. 0·05 c.c. of blood, taken from the point of the finger by suction with a capillary pipette, is placed with a little water in a crucible, evaporated to dryness and incinerated. 0·1 gramme of dry powdered hydrogen potassium sulphate is then added, and the whole fused. After cooling, the contents of the crucible are dissolved in hot water, and the iron determined colorimetrically. H. H. B. S.

INORGANIC ANALYSIS.

The Estimation of Oxygen in Air, etc. D. Albert Kreider. (*Zeit., anorg. Chem.*, xiii., 6, 418-426.)—A measured volume of air is shaken up with a strong solution of hydriodic acid, nitric oxide being present. The acid is then neutralized with potassium bicarbonate, and the liberated iodine titrated with a decinormal solution of arsenious acid. Care must be taken that all the solutions used are free from dissolved oxygen. 100 c.c. of air is a convenient quantity to work upon, and for this 15 c.c. of nitric oxide are sufficient. Experiments—seventeen in all—made by the author gave percentages of oxygen in air varying from 20·91 to 21·19. Two comparative estimations by the pyrogallol method gave 20·93 and 20·88 per cent.

The amount of oxygen dissolved in water may be determined by this method by means of the apparatus shown in the accompanying figure. The large bulb holds about 300 c.c. when filled to the mark *C*. The bulb *a* has a capacity 30 c.c. The capacity of the larger bulb must be accurately determined. The whole apparatus is completely filled with the water to be examined. Nitric oxide is then passed in by the tube *d*, the tap *b* being opened until the water sinks to the mark *C*; 2 c.c. of strong hydrochloric acid are run in through the tube *h*, the tap *d* being opened sufficiently to allow the acid to enter, and finally a solution of potassium iodide is run into the apparatus in the same way. After thoroughly shaking up the contents, the liberated iodine is titrated as described above.



The whole operation need not take more than ten minutes to carry out. The mean of twelve experiments gave 6·022 c.c. of oxygen as the amount of this gas which was dissolved in 1,000 c.cm of distilled water, at 20° C. and 760 mm. pressure.

The above method is applicable for the determination of the amount of carbonic acid dissolved in water. W. P. S.

A Modification of the Gunning Method for Nitrates. J. Fields. (*Jour. Amer. Chem. Soc.*, 1896, xviii., 1102-1104.)—The object of the modification described below is to reduce the number of reagents required, and to largely prevent the tendency to froth, which is often very troublesome in the official Gunning method.

The weighed material is gently heated with 30 c.c. of sulphuric acid containing one gramme of salicylic acid, and, while warm, six or seven grammes of potassium sulphide are added little by little, the flask being shaken after each addition. It is then placed over a low flame and the heat rapidly increased until the mixture boils. No further attention is then required, and the digestion is usually complete at the end of an hour. The potassium sulphide does double duty, it first acts as a reducing agent in place of sodium thiosulphate, and after being converted into potassium bisulphate, acts as that salt does in the original method.

The average difference between the results given by the official method and this modification when tried on sixty samples of fertilizers was 0·02 per cent., the modification giving the higher figures. C. A. M.

Determination of Oxide of Iron and Alumina in Phosphates. F. Lichtschlag. (*Chem. Zeit.*, xxi., No. 28, 264.)—Two grammes of the finely-powdered phosphate and about 8 grammes of sodium carbonate are fused together in a platinum crucible. The fused mass is dissolved in water, with the aid of 10 to 15 c.c. of dilute hydrochloric acid to remove the last portions from the crucible, and a large quantity of hydrochloric acid is then added. Altogether 100 to 125 c.c. hydrochloric acid of specific gravity 1·095 are required. The acid liquid is digested for ten minutes at 50° to 60° C., transferred to a 200 c.c. flask, neutralized with a strong solution of sodium hydrate, heated nearly to boiling, 30 c.c. of a 20 per cent. sodium hydrate solution added, and the whole digested for fifteen minutes with frequent shaking. After cooling, the flask is made up to the mark, the contents well mixed, and 50 c.c. of the filtered solution taken for the determination of the alumina.

The precipitate upon the filter is dissolved in hydrochloric acid, and the iron determined by reducing with zinc and titrating with permanganate, observing the precautions recommended by Fresenius for cases in which hydrochloric acid is present.

For the determination of the alumina, the 50 c.c. are acidified with hydrochloric acid, evaporated to dryness, taken up with a little hydrochloric acid, diluted with hot water, and filtered and washed into a beaker. The filtrate is heated to boiling, made slightly alkaline with ammonia, the precipitate allowed to settle, filtered off, and washed with hot water until the washings are free from chlorine. The filter and precipitate are then ignited and weighed. The ignited precipitate consists of $AlPO_4$. It is advisable to add a little sodium phosphate before the precipitation with ammonia. H. H. B. S.

Determination of Chromium in Ferro-Chrome and Chrome-Steel. J. Spüller and A. Brenner. (*Chem. Zeit.*, 1897, xxi., 3.)—This process has been employed in the Kladno laboratory for some years, and if the following details are attended to, it will be found very speedy as well as accurate. 0·35 gramme of the finely-powdered

sample of ferro-chrome is mixed with 2 grammes of dry powdered caustic soda in a hemispherical silver basin, and 4 grammes of sodium peroxide are sprinkled on it The vessel is heated until the mass begins to melt, the flame is then instantly removed, and the whole stirred together with a silver spatula. When the violence of the reaction has passed, the burner is again applied, 5 grammes of peroxide are added after ten minutes' fusion, and the temperature then raised till the mass becomes perfectly fluid. In half an hour 5 grammes more of peroxide are introduced, and after a further twenty minutes the oxidation will be found complete. A final addition of 5 grammes of peroxide is made to render the melt more easy of solution, and as the basin cools it is constantly stirred in order to spread the contents over its surface. The vessel is then dropped into a covered porcelain basin containing water, and in a very few minutes all the soluble matter will be extracted. After subsidence the liquid ought to be yellow; but if it be coloured green or red owing to the presence of sodium manganate or permanganate, it may be treated with more peroxide; whilst if this prove ineffectual, a few c.c.'s of a strong permanganate solution should be run in, and the whole reduced afresh. A current of carbon dioxide is then passed through the hot liquid for thirty minutes, and after cooling it is made up to 1 litre, and the chromic acid determined in 250 c.c by Schwarz's process.

In the case of chrome (or chrome-nickel) steels, 2 grammes are dissolved in 20 c.c. of strong hydrochloric acid, 10 c.c. of 1:1 sulphuric acid added, the solution evaporated, and the excess of acid removed. The residue is intimately mixed with 2 grammes of caustic soda as before, and 5 grammes of sodium peroxide sprinkled on top. The mass is heated cautiously till the sulphates are decomposed and it begins to cohere. Three more additions of 5 grammes each of peroxide are made at intervals of twenty minutes; and the melt is finally treated as above, except that it is only dissolved in 500 c.c. of water, and 250 are used for titration. When the amount of chromium is small, this is carried out by Zulkowsky's process as follows: The solution is placed in a tall beaker, 10 c.c. of 10 per cent. potassium iodide added, and pure HCl (sp. gr. 1·12) run in till the liquid is just acid. Twenty c.c. of bichromate containing 0·9833 gramme (= 0·35 gramme Cr) per litre are treated in a precisely similar manner, and after both liquids have been allowed to stand in the dark for fifteen minutes, they are titrated side by side with $\frac{N}{50}$ thiosulphate. F. H. L.

LEGAL.

IMPORTANT DECISIONS IN THE HIGH COURT OF JUSTICE.

(Reprinted from the "Times" of May 8, 1897.)

ARSENICAL SOAP.

HOUGHTON *v.* TAPLIN.

This was a case stated by the justices of Richmond upon dismissing a summons under the Sale of Food and Drugs Act, 1875.

Mr. A. Glen appeared for the appellant, and Mr. Lawless for the respondent.

An information was laid against the respondent charging him with selling to the prejudice of the appellant a drug, to wit, arsenical soap, which was not of the nature, substance, and

quality of the article demanded, contrary to Section 6 of the Sale of Food and Drugs Act. The appellant asked for arsenical soap, and he was supplied by the respondent with a tablet of Dr. Mackenzie's arsenical toilet-soap. The soap contained no arsenic. The magistrates dismissed the information, holding that though arsenical soap was a drug within the meaning of the Act, yet as the soap in question contained no arsenic it was not a drug, and no offence had been committed.

Mr. GLEN contended that the subject of the sale was a drug, though it was proved that there were no medicinal properties in the article delivered.

Mr. JUSTICE WRIGHT: Was not the article demanded a compounded drug and within the third exception in Section 6?

Mr. GLEN: It was a compounded article, but not a compounded drug.

The COURT dismissed the appeal, Mr. JUSTICE HAWKINS on the ground that the soap was not a drug, and Mr. JUSTICE WRIGHT on the ground that the soap was a compounded drug within the meaning of Sub-section 3.

FALSE WARRANTY.

DERBYSHIRE *v.* HOULISTON.

This was an appeal, by case stated, from a conviction under Section 27 of the Sale of Food and Drugs Act, 1875.

Mr. C. A. Russell, Q.C., and Mr. F. H. Mellor appeared for the appellant; and Mr. Lawson Walton, Q.C., and Mr. Reginald Brown for the respondent.

An information was laid against the appellant charging him with giving a false warranty in writing to one Martin Hopkins, in respect of certain butter sold by him and subsequently sold by Martin Hopkins to the respondent, which was not of the nature, substance, and quality demanded by the respondent, contrary to Section 27 of the Sale of Food and Drugs Act, 1875.

The respondent bought the butter in question from Martin Hopkins on September 16, 1896. It was marked "Pure butter, 10d.," but was subsequently discovered to contain 23 per cent. water. Martin Hopkins was proceeded against, but on proof that he had purchased the butter from the appellant on September 8 with a written warranty to the effect that it was "pure butter" the summons was dismissed. The appellant showed that he had purchased the butter on August 22 from Martin Malony with a written warranty that it was "guaranteed pure Irish butter." The magistrate before whom the information was heard convicted the appellant, being of opinion that it was not necessary to prove that the appellant, when he gave the warranty to Hopkins, knew that it was false, and that on the facts proved the appellant was not entitled to the protection afforded by Section 25 of the Act. It was argued on behalf of the respondent that an unlawful intention was not part of the offence.

Mr. JUSTICE HAWKINS, in allowing the appeal and remitting the case to the magistrate, said that in order to constitute the offence with which the appellant was charged it was necessary that there should be an unlawful intention. It was clear that that was the intention of the Act, because the offence was associated in the same section with three offences in which it was clear a guilty knowledge was implied. It was contended that the offence of giving a false warranty ought to be so construed as to cover the case of a person who had no knowledge that the warranty was false. It was monstrous to say that a person was liable to a fine of £20 when he believed the warranty he was giving to be true. It was true that there were cases—for instance, under the Explosives Act—where knowledge was presumed, unless the contrary were shown, but that was never done, except in cases where the Legislature had very clearly indicated that it intended to punish a man who was not morally guilty.

Mr. JUSTICE WRIGHT said that he was of the same opinion. He referred to Sherras *v.* De Rutzen, a case in which he had cited authorities in support of the proposition that a *mens rea* was essential in every offence, unless it were clearly displaced by statute. That guilty knowledge was a necessary element in the offence with which the appellant was charged was indicated by the use of the word "false," which implied a knowledge of the falsity of the warranty; it was further indicated on the principle of *noscitur a sociis* by the offence being imbedded in the same section with others, all of which implied guilty knowledge.

ERRATUM.—The second paragraph which appears on page 124 of Mr. Stoddart's paper in the previous number of THE ANALYST is misplaced. It should be read in connection with the table on page 120, to which it refers.

THE ANALYST.

JULY, 1897.

OBITUARY.

DR. C. REMIGIUS FRESENIUS.

It is with the deepest regret that we have to record the death of Dr. R. Fresenius, the Nestor of analytical chemists. Suddenly struck down by apoplexy, he peacefully passed away at midnight, June 10-11, at his home in Wiesbaden, whence for just upon half a century the results of his incessant chemical labours have flowed. Literarily and scientifically active to the very day of his death—for his work was to him ever a labour of love, and the thought of rest from work hardly had any existence with him—we have lost in Fresenius one of the few remaining eminent men who connect the present generation of chemists with the period of Liebig's greatest activity and brilliancy.

Fresenius was born at Frankfort-on-the-Maine in 1819, and was therefore in his seventy-ninth year at the time of his death. He was educated at the Gymnasium at Frankfort, and in 1836 entered as apprentice the pharmaceutical establishment of Stein, leaving there in 1840 for the University of Bonn. As a student at Bonn, he felt the necessity for a more systematic scheme of qualitative analysis than was in existence at that date, and in order to fill this deficiency he worked out such a scheme. This was published in book form in 1841 by Henry and Cohen, of Bonn, under the title, "Anleitung zur qualitativen Chemischen Analyse." It was the commencement of those books on analysis, translations of which have appeared in very numerous languages, which have made Fresenius' name a household word among chemists. In 1841 he left Bonn for Giessen, where he at once became assistant to Liebig, and there conducted the laboratory course in elementary inorganic analysis. Two years later Fresenius became "Privatdocent" at the University of Giessen, remaining there till September, 1845, when he received a call to the Chair of Chemistry, Physics, and Technology in the Agricultural Institute near Wiesbaden. The means of the Institute in question were exceedingly limited, and hardly allowed any practical chemical work to be carried on. Fresenius therefore, at the commencement of 1848, established the private laboratory with which his name is inseparably connected. Only the small number of five pupils worked with him during the first year, but their number rapidly increased, for about fifty joined him in 1855, and this necessitated the engagement of a staff of lecturers, among whom

C. Neubauer, the author of the well-known work on the analysis of urine, was the most prominent.

The teaching of Fresenius and his staff was officially recognised by the Government of the Duchy of Nassau (one of the small States absorbed by Prussia in 1866), and a financial subsidy was paid him by the State. An official agricultural and œnological laboratory was in later years combined with the establishment, which gradually became a centre of analytical research; while commercial and technological chemical investigations, touching on all branches of industry, kept a large staff of assistants employed. Though the later development of the chemical laboratories at the various German Universities largely removed the need for a tutorial establishment founded upon private enterprise, the large Institution, over all the branches of which Fresenius unceasingly watched, continued to do exceedingly good work, and to prosper scientifically and financially. When to all this is added the large amount of labour involved in the management of the journal, the *Zeitschrift für Analytische Chemie*, established by Fresenius; in the frequent issue of new editions of his works, and in the supervision of chemical manufactories, it will be seen that his was no ordinary energy. He also took an active interest in the politics of the day, and for the last few years of his life held the chairmanship of the municipality of Wiesbaden. The method of his work from morning till night, however, enabled him for just upon fifty years to fulfil his numerous self-imposed duties, and to make himself a power in chemical science—at least, in that one branch out of which, scientifically, he never stepped.

But a short time ago he expressed to the writer of this notice the hope that on the completion of the half-century of the conduct of his laboratory—which would have happened during the coming spring—he might fittingly retire.

Looking over the scientific results of his life, it can safely be asserted that no chemist of modern times since the death of Rose has left a deeper imprint upon analytical chemistry than Fresenius has. To give a list alone of the original papers written by him would cover many pages, for they range throughout the whole territory of inorganic chemistry. The most important among them are those earlier methods, such as that worked out with Will in 1843 for the valuation of the carbonates and of manganese peroxide; the use of potassium cyanide in analytical work; the differentiation of the arsenic and antimony mirrors in Marsh's process; the cyanide method of detecting arsenic in forensic cases (with Babo); his numerous contributions to the analysis of phosphates, of acetates, and of sulphates.

Much of his most careful work was done in connection with the publication of his books. He made it his duty to test almost every method of analysis which he included in his "Text-book of Quantitative Analysis"; and he conscientiously published most of the data he thus obtained. If, in this his desire to give exact information, he was often too full of detail for modern scientific taste, or if, in his anxiety to reach the highest possible accuracy, he carried decimal points somewhat farther than necessary, this does not detract from, but rather adds to, the impression which his honesty of purpose is bound to make. As in these days of specialization one chemist may find interest only in the azo-bodies, another in the derivatives of naphthalene, so Fresenius was interested only in analytical methods, and

was opposed, actively at first, passively in later years, to whatever appeared to him to stand in the way of analytical science. Thus, the doubling of many of the old equivalents, rendered necessary by the adoption of the laws of Avogadro and of Dulong and Petit, was utterly uncongenial to him, and he saw the decay, nay, the rout, of the dualistic conceptions with dismay and sorrow, and stood out, longer almost than any other writer, for the older notation.

His undying merit is, without doubt, the writing of his two works on Analytical Chemistry, in the first place, and the establishment and conduct of the *Zeitschrift für Analytische Chemie* in the second. His "Qualitative Analysis" has gone through sixteen German editions and the "Quantitative" through six. Translations exist in a multitude of languages, and every chemical laboratory in the world works according to the information contained in these volumes. In inorganic analysis they are the most trustworthy guides that were ever written.

Personally, Fresenius was of a most kindly and genial disposition; he took a loving interest in every one of his students, and inspired many of them with the enthusiasm which he so obviously felt. Not only to them will he remain in loving memory, but as long as analytical work is carried on in chemical laboratories, and as long as his methods of analysis are used in industry and in research, will the name of C. Remigius Fresenius be honoured as that of one of the worthies of chemical science.

O. H.

PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS.

THE monthly meeting of the Society was held on Wednesday evening, June 2, in the Chemical Society's Rooms, Burlington House, the President (Dr. Bernard Dyer) occupying the chair.

The minutes of the previous meeting were read and confirmed.

The PRESIDENT announced that Dr. W. J. Russell, F.R.S., had been nominated by the Council for election as an Honorary Member of the Society. Dr. Russell, as President of the Institute of Chemistry, had worked very hard during the last three years, and had earned the thanks of the profession at large, and the Council was anxious that the Society should recognise his services by electing him an Honorary Member. (Applause.)

The following papers were read: "Note on the Presence of Heavy Metals in Cheese," by Alfred H. Allen and F. Hudson Cox; "On the Structure of the Fat Globules in Cows' Milk," by Professor V. Storch, Copenhagen (communicated by H. Faber); "On the Indirect (Tabarie's) Method for the Estimation of Alcohol," by Norman Leonard, B.Sc., and Harry M. Smith; "The Determination of Soluble Proteids by Chlorine Precipitation," by S. Rideal, D.Sc., and C. J. Stewart.

The following were taken as read: "Note on Commercial Ground Cassia," by W. F. Keating Stock; "Note on the Bacteriolysis of Gelatin," by S. Rideal, D.Sc., and Ronald Orchard.

Mr. Henry Bradford (assistant to Mr. Clayton) was elected an Associate of the Society.

Summer Meeting.

The President said that as, on account of the forthcoming Jubilee festivities, the arrangements of both town and country members during the next few weeks might be expected to be somewhat disorganized, the Council deemed it advisable that the summer meeting (which in the ordinary course would be held in July) should be postponed. It had been decided that the first meeting after the summer recess should this year be held in October instead of November, and that on either the day before or the day after that meeting there should be held what under ordinary circumstances would be the summer meeting.

NOTE ON COFFEE-PALACE COFFEE INFUSIONS.

By E. G. Clayton.

(*Read at the Meeting, March* 3, 1897.)

Eleven samples of coffee infusion supplied in coffee-palaces and similar establishments in London were lately examined by the author, with the assistance of Mr. Henry Bradford. The samples contained caffeine in proportions varying from 0·014 to 0·039 per cent., the average being 0·026. The actual figures obtained were 0·014, 0·015, 0·016, 0·017, 0·027, 0·028, 0·031, 0·033, 0·033, 0·036, and 0·039.

As a 10 per cent. infusion of coffee was found to contain nearly 0·12 per cent. of caffeine, it follows that the strength of these coffee infusions varied from about 1 to 3 parts of coffee to 100 parts of water, the average being about 2 parts per 100 of water.

Since in nearly every case the infusion was already mixed with milk and sugar, there was no method of arriving at the average probable proportion of chicory present.

ON THE VALUE OF THE NITROGEN FACTOR IN THE ANALYSIS OF DECOMPOSED MILK.

By Alfred Smetham, F.I.C., F.C.S., and J. B. Ashworth, F.C.S.

(*Read at the Meeting, May* 5, 1897.)

Having in the latter part of last year under examination a sample of milk which had undergone decomposition, it occurred to one of us that a more reliable opinion of the original composition could be obtained by estimating the nitrogen and ash, and thus calculating the original percentage of casein and albumen and ash present in the sample, than by attempting to estimate the total solids-not-fat, and calculating by any system of correction back to the fresh condition of the milk. In investigations which we had conducted upon the cheese-making industry of Cheshire on behalf of the Board of Agriculture, we had been struck by the comparatively constant contents of casein and albumen in the mixed milk of herds from various sources and at different

times; and as our experiments had further shown that the production of lactic acid commences at a very early stage of decomposition, it seemed pretty clear that any ammonia formed by fermentation of the nitrogen compounds would be fixed, and as long as the milk remained acid an estimation of nitrogen (unless free nitrogen were evolved) would yield results identical—except so far as there was loss of weight of the sample—with those obtained in the analysis of the fresh milk.

In this connection the following figures, obtained by titrating milk with KHO, using phenolphthalein as indicator, may prove interesting:

		Acidity per cent. as Lactic Acid.
Milk direct from cow		·24
,, ,, (after boiling)		·11
Mixed evening milk from vat (in evening)		·17
,, ,, ,, (next morning)		·19
(Average of 91 determinations.)		
Sept. 8. Milk, original from vat		·15
Milk kept in dairy 2 days		·70
,, ,, 3 ,, ...		·97
,, ,, 7 ,, ...		·97
,, ,, 16 ,, ...		1·08
Sept. 23. Milk put up 7 p.m.		·17
Sept. 24. ,, ,, 6.30 a.m.		·18
,, ,, ,, 2.30 p.m.		·19
,, ,, ,, 11.30 p.m.		·20
Sept. 25. ,, ,, 10 a.m.		·22
Sept. 26. ,, ,, 10 a.m.		·88
Sept. 29. ,, ,, 10 a.m.		1·15

The sample of milk marked No. 8 in the tables, after being kept for thirteen weeks gave, on distillation with magnesia, ammonia = ·055 per cent., and it will be seen that up to that time the lactic acid is much in excess of the ammonia produced.

To ascertain to what extent changes in the nitrogen contents of milk took place during keeping, we started a series of experiments, the results of which we have now the honour to present to you.

The method adopted was as follows: Nine milks from various sources, of the purity of which there was no doubt, were obtained and analysed, the total solids dried to constancy at 100° C, the fat estimated by Adams' process, using Schleicher and Schüll's fat-free paper (which gave only 0·0005 gramme of residue on extraction with ether), the nitrogen estimated in duplicate by the Kjeldahl-Gunning-Arnold process, in exactly the manner described by our President (*Journal, Chemical Society*, lxvii., 811), the water of the milk being evaporated over a low flame after the addition of the sulphuric acid, the nitrogen × 6·33 stated as casein and albumin. The milk-sugar, etc., was obtained by difference.

At the same time that the quantities for the analyses were weighed out, six portions of about 10 grammes were accurately weighed in sterilized glass bottles of about 15 c.c. capacity, and these were corked, kept at the ordinary temperature of the laboratory, and the nitrogen estimated at the expiration of different periods. The following is a description of the sources of the samples of milk treated in this way:

1. Milk from a mixed herd of cows, delivered December 7, 1896.
2. Morning's milk from Cheshire Dairy Institute, December 10, 1896, being the mixed milk of 28 shorthorn and 2 Jersey cows; 20 calved March and April, 1896, and the other 10 since the middle of October. Daily rations: 4 lb. decorticated cotton-cake, 2 lb. maize meal, 2 lb. bran, and hay *ad lib.*
3. Evening's milk from above herd, December 9, sampled in morning of December 10. The cream evidently had not been thoroughly mixed with the milk.
4. Milk from 4 Dutch cows on Lord Egerton's estate, December 18, 1896; calved respectively October 30, September 31, September 28, and August 20. Food: Indian meal, linseed cake, bran, meal, hay, and mangolds.
5. Milk from 4 dairy shorthorns on Lord Egerton's estate, December 18, 1896; calved respectively August 30, August 27, June 26, and June 15. Food as No. 4.
6. Milk from 4 Jersey cows on Lord Egerton's estate, December 18, 1896; calved respectively October 24, September 15, September —, and September —. Food as No. 4.
7. Milk from 4 Kerries on Lord Egerton's estate, December 18, 1896; calved respectively August 16, June 30, February 26, and February 20. Food as No. 4.
8. Mixed morning milk from farm of Cheshire Dairy Institute, January 25, 1897. Herd and feeding same as No. 2.
9. Mixed evening milk from farm of Cheshire Dairy Institute, January 24, 1897; taken same evening. Herd and feeding same as No. 2.

The table on next page gives the original composition of the samples, the percentage composition of the solids-not-fat, the contents of nitrogen—stated as casein and albumin—and the percentage loss of weight in each sample at the time the estimation was made.

From these results it is evident that, under the conditions obtaining in these experiments, the loss of nitrogen on keeping the milk for periods up to twenty-six weeks is nil, or, at all events, does not exceed what may reasonably be deemed errors of manipulation; and it seems to us, therefore, that so long as a sample of milk has an acid reaction, an estimation of the nitrogen enables the analyst to infer with a probability amounting almost to certainty what was the original contents of casein and albumen in the sample, the only uncertainty being the loss of weight the sample has undergone on keeping.

On finding in the earlier stages of our investigation that our presumption that the nitrogen contents of a sample of milk remained constant on keeping, we obtained other samples from different sources, with a view of ascertaining to what extent the percentages of the albumen and casein varied in milk from different sources, and to what extent these enabled the analyst to infer adulteration or otherwise.

The samples examined were as follows:

10. Milk from a Liverpool dairy, stall fed, February 16, 1897. Feeding unknown.

	1. Milk from a Dairy, Rock Ferry.	2. Morning's Milk from Dairy Institute, Worleston, Cheshire, 28 Shorthorns, 2 Jerseys, Dec. 10, 1896.	3. Evening's Milk from Dairy Institute, Worleston, 28 Shorthorns, 2 Jerseys, Dec. 9, 1896.	4. Milk from 4 Dutch Cows.	5. Milk from 4 Dairy Shorthorns.	6. Milk from 4 Jerseys.	7. Milk from 4 Kerries.	8. Morning's Milk from Dairy Institute, Worleston, 28 Shorthorns, 2 Jerseys, Jan. 25, 1897.	9. Evening's Milk from Dairy Institute, Worleston, 28 Shorthorns, 2 Jerseys, Jan. 24, 1897.
Water	87·43	87·56	89·68	88·55	87·23	84·55	85·64	87·81	87·30
Fat	3·35	4·05	1·80	3·25	4·07	6·21	5·05	3·77	4·41
*Casein and albumin (N × 6·33)	3·48	3·37	3·40	2·93	3·43	3·93	4·15	3·26	3·22
Milk, sugar, etc.	4·94	4·24	4·32	4·56	4·50	4·51	4·40	4·39	4·36
Ash	·80	·78	·80	·71	·77	·80	·76	·77	·71
	100·00	100·00	100·00	100·00	100·00	100·00	100·00	100·00	100·00
*Containing nitrogen	·549	·532	·537	·463	·542	·621	·655	·515	·509
Solids-not-fat	9·22	8·39	8·52	8·20	8·70	9·24	9·31	8·42	8·29
Percentage composition of solids-not-fat:									
Casein and albumin	37·42	40·17	39·91	35·73	39·43	42·53	44·58	38·72	38·84
Milk-sugar, etc.	53·90	50·53	50·70	55·61	51·72	48·81	47·26	52·14	52·59
Ash	8·68	9·30	9·39	8·66	8·85	8·66	8·16	9·14	8·57
	100·00	100·00	100·00	100·00	100·00	100·00	100·00	100·00	100·00
After 1 *week*:									
Loss per cent.	·10	·12	—	·12	·14	·20	·06	—	—
Casein and albumin	3·46	3·31	—	2·88	3·42	3·93	4·18	—	—
After 2 *weeks*:									
Loss per cent.	·40	·12	—	·40	·23	·21	·18	·10	·06
Casein and albumin	3·46	3·42	—	3·02	3·52	3·89	4·20	3·22	3·22
After 3 *weeks*:									
Loss per cent.	·39	1·00	—	—	—	—	—	—	—
Casein and albumin	3·52	3·46	—	—	—	—	—	—	—
After 4 *weeks*:									
Loss per cent.	·36	2·57	·33	·33	·44	·36	·33	—	—
Casein and albumin	3·55	3·29	3·44	2·89	3·51	4·01	4·21	—	—
After 5 *weeks*:									
Loss per cent.	—	—	—	—	—	—	—	·24	1·21
Casein and albumin	—	—	—	—	—	—	—	3·15	3·19
After 7 *weeks*:									
Loss per cent.	—	—	—	·37	·22	·75	·42	—	—
Casein and albumin	—	—	—	2·88	3·39	3·86	4·05	—	—
After 8 *weeks*:									
Loss per cent.	—	—	—	—	—	—	—	·17	·82
Casein and albumin	—	—	—	—	—	—	—	3·11	3·15
After 9 *weeks*:									
Loss per cent.	·87	·41	·81	—	—	—	—	—	—
Casein and albumin	3·46	3·30	3·36	—	—	—	—	—	—
After 12 *weeks*:									
Loss per cent.	1·57	—	—	—	—	—	—	—	—
Casein and albumin	3·53	—	—	—	—	—	—	—	—
After 13 *weeks*:									
Loss per cent.	—	·62	·50	—	—	—	—	1·01	—
Casein and albumin	—	3·48	3·45	—	—	—	—	3·11	—
After 14 *weeks*:									
Loss per cent.	—	—	—	—	—	—	—	—	·51
Casein and albumin	—	—	—	—	—	—	—	—	3·15
After 19 *weeks*:									
Loss per cent.	—	—	—	·49	·44	1·13	·70	—	—
Casein and albumin	—	—	—	2·84	3·53	4·00	4·15	—	—
After 21 *weeks*:									
Loss per cent.	—	—	—	—	—	—	—	·61	·62
Casein and albumin	—	—	—	—	—	—	—	3·16	3·28
After 26 *weeks*:									
Loss per cent.	—	—	—	1·40	·50	1·42	1·33	—	—
Casein and albumin	—	—	—	2·81	3·39	3·86	4·15	—	—

11. Milk from another town dairy, February 23, 1897.
12. Milk delivered to a house at Crosby, March 9, 1897. Origin unknown.
13. Milk from a Cheshire farm near Warrington, March 10, 1897. Cows fed with clover hay, Indian corn, oats, swedes, and mangolds.
14. Mixed milk from 4 dairy shorthorns, April 1, from Lord Egerton's home farm, Tatton Park:

	Yield of Milk.		Calved.
	Morning. qts.	Evening. qts.	
"Trinket" ...	7 ...	6 ...	August 30
"Kirby Belle" ...	4½ ...	4 ...	August 27
"Cuckoo" ...	7 ...	5 ...	February 19
"Chaffinch" ...	6 ...	4½ ...	February 20

Daily rations: 2½ lb. Indian meal each a meal.
1 lb. cake each a day.
½ lb. bran each a day.
3 fodderings (with meadow hay).
2 mangolds each a day.

15. Mixed milk from 4 Dutch cows, April 1, 1897:

	Yield of Milk.		Calved.
	Morning. qts.	Evening. qts.	
"Checkmate" ...	8½ ...	7 ...	November 11
"Dutch Maid" ...	7 ...	5½ ...	November 20
"Chessboard" ...	8 ...	7 ...	December 23
"Stella" ...	6 ...	5 ...	November 1

Daily rations same as No. 14.

16. Mixed milk from 4 Kerries, April 1, 1897:

	Yield of Milk.		Calved.
	Morning. qts.	Evening. qts.	
"Kerry A" ...	5½ ...	4 ...	March 4
"Kerry K" ...	4½ ...	3 ...	February 14
"Killala" ...	5 ...	3½ ...	March 5
"Kerry No. 1" ...	3½ ...	2½ ...	January 29

Daily rations: 1¾ lb. Indian meal each a meal.
½ lb. cake each per day.
½ lb. bran each per day.
2 mangolds each per day.
3 fodderings (of meadow hay).

17. Mixed milk from 4 Jersey cows, April 1, 1897:

	Yield of Milk.		Calved.
	Morning. qts.	Evening. qts.	
"Phyllis" ...	5 ...	4 ...	October 1
"Wallflower" ...	2½ ...	2 ...	October 1
"Buttercup" ...	4½ ...	4 ...	October 24
"Tulip" ...	3½ ...	3 ...	October 1

Daily rations same as No. 14.

18. Milk taken at Stockport by inspector under Sale of Food and Drugs Act, April 13, 1897.

19. Ditto, ditto (another sample), April 13, 1897.

On analysing in the same manner as before the accompanying results were obtained:

	10. Liverpool Dairy. Town fed.	11. Liverpool Dairy. Town fed.	12. Delivered at Crosby.	13. Cheshire Farm, near Warrington.	14. From Dairy Shorthorns.	15. From 4 Dutch Cows.	16. From 4 Kerry Cows.	17. From 4 Jersey Cows.	18. From Stockport.	19. From Stockport.
Water	86·62	88·11	87·21	87·41	87·81	88·76	86·29	84·15	86·86	87·19
Fat	4·92	3·10	4·03	3·72	3·69	3·21	5·02	6·35	4·39	4·30
*Casein and albumin (N × 6·33)	3·68	3·46	3·78	3·65	3·33	2·74	3·33	4·04	3·51	3·28
Milk-sugar, etc.	4·01	4·56	4·20	4·40	4·44	4·57	4·65	4·72	4·47	4·36
Ash	·77	·77	·78	·82	·73	·72	·71	·74	·77	·87
	100·00	100·00	100·00	100·00	100·00	100·00	100·00	100·00	100·00	100·00
*Containing nitrogen	·581	·546	·597	·576	·526	·434	·525	·639	·554	·518
Solids-not-fat	8·46	8·79	8·76	8·87	8·50	8·03	8·69	9·50	8·75	8·51
Percentage composition of solids-not-fat:										
Casein and albumin	43·50	39·36	43·15	41·15	39·18	34·12	38·32	42·52	40·11	38·54
Milk-sugar, etc.	47·40	51·87	47·95	49·60	52·23	56·91	53·51	49·69	51·09	51·24
Ash	9·10	8·77	8·90	9·25	8·59	8·97	8·17	7·79	8·80	10·22
	100·00	100·00	100·00	100·00	100·00	100·00	100·00	100·00	100·00	100·00

From the whole of the analyses it will be noticed that, with the exception of the milk from the Dutch cows, the lowest percentage (3·22) of albuminoids is in No. 9—a result slightly above that suggested by Mr. Richmond as a limit (ANALYST, xxii. 94). The average percentage of albuminoids is 3·466. If the milk from the Dutch (which gives in both cases milk of a very poor character), the Kerry, and Jersey cows, which do not as a rule form any appreciable proportion of the herds kept for the production of milk for towns' supplies, be omitted, the average percentage is 3·448.

The average percentage of milk-sugar obtained by difference is 4·45, with 4·01 as a minimum, and 4·97 per cent. as a maximum.

From the results of the analyses it will be observed that the variations in the percentages of casein and albumin are greater than those of the solids-not-fat, and consequently the nitrogen is a less satisfactory factor in determining adulteration than the sum of the total solids-not-fat. Nevertheless, as it enables the analyst to calculate with a great degree of accuracy the original contents of the casein and albumin, it offers, in conjunction with the ash, a means of control in the analysis of decomposed milks probably more accurate than any system depending upon the estimation of the total solids and a correction for decomposition, when the nature of the decomposition is unknown.

Discussion.

The President said that this contribution from Mr. Smetham and Mr. Ashworth came as a matter of considerable interest just after the suggestion lately made by Mr. Richmond, who did not know at the time that this work was in progress. One point that was brought out in these analyses was a point which Mr. Richmond had emphasized before, viz., that the most constant figure in a milk analysis was the milk-sugar. Unfortunately, however, it was the item which underwent most change on keeping. Working out the ratio of nitrogen to non-fatty solids in these analyses, he noticed that the variation was considerable, being from 1 : 14 in the case of the Kerry cows to 1 : 18 in the Dutch cows, the lowness of the non-fatty solids in the latter case probably being partly due to a deficiency of nitrogenous constituents. These Dutch cows occasionally caused trouble. He had met with cases himself in which farmers had got into trouble by relying upon Dutch cows, or by selling the milk of Dutch cows which they happened to possess, without mixing it in with the rest of the milk from a miscellaneous herd. Mr. Allen had written to him expressing regret at being unable to attend the meeting, and mentioning that he had a high opinion of the value of the nitrogen factor in the analysis of decomposed milks, and saying that in the forthcoming volume of his work on "Commercial Organic Analysis" he was suggesting a limit of 0·5 per cent. of nitrogen, which was also the figure proposed by Mr. Richmond.

Mr. Richmond said he considered the paper a very valuable one. He believed that this was the first series of systematic nitrogen determinations in milk after keeping that had yet been published, and he might say that the results agreed entirely with his own experience, although he could not claim to have made a systematic series of determinations. The most important point in the paper seemed to him to be the low proportion, both of solids-not-fat and of casein and albumin, yielded by the Dutch cows. He had had no opportunity for noticing this in his own experience, for he had not analysed any samples taken from those cows alone, which formed an infinitesimal minority among the herds from which the milk that he examined was drawn. It seemed to him, however, that the milk of Dutch cows was more or less abnormal. Similar results to those recorded by the authors had been observed in the United States at the New Jersey Experiment Station in the case of cows of a practically identical breed. The nitrogen in many cases fell distinctly below 0·5 per cent., the ash below 0·7, and the solids-not-fat frequently as low as 8 per cent. He thought that, in considering any limits that might be fixed for milk, these cows must be taken into account. It would hardly do to declare that the product of Dutch cows was not milk. The farming interest of the country would claim to be allowed to keep any particular breed of cows that they chose, and also that the normal product of these animals, whatever their breed might be, was entitled to be considered as milk. It would be for the Society of Public Analysts, or any other body that might have to fix limits for milk, to consider how far the Dutch cows would affect the figures that were chosen. In the first portion of the paper some figures were given as to the acidity of milk, estimated as lactic acid, and it was stated that milk containing 0·22 per cent. of acidity as lactic acid showed, after heating to 100° C., a

diminution of the lactic acid to 0·11 per cent. He would like to ask the authors how they accounted for this. He had himself found practically the same thing, and his own idea was that the phenomenon was to be largely accounted for by the deposition of acid calcium salts, to which a large proportion of the acidity of milk was due. He would like to know whether the authors agreed with him in this, or whether they had any other explanation to suggest. He would also like to draw attention to the fact that, in estimating the acidity of sour milk, when phenol-phthalein was used, the dissolved carbon dioxide was estimated, and the acidity, if calculated to lactic acid, would be largely in excess of the true lactic acid present. It was by no means uncommon for a milk which had been kept some time and which was distinctly sour, to show as much as 2 per cent. of lactic acid, which he believed was a quantity that was not formed by the lactic ferment; the real lactic acid would in such a case be perhaps about half this amount.

Mr. R. W. Woosnam remarked that the nitrogen determinations mentioned in the paper had all been made on samples carefully weighed out while still fresh. He did not know how the authors would deal with a sample that had been bottled up for about six weeks, the condition of which would probably be very bad indeed. He supposed they would follow the usual plan of neutralizing with caustic soda and shaking vigorously, but it would be very difficult to get a fair sample at all in such a case.

Mr. Richmond observed that an excellent method for mixing up decomposed milks was described in Dr. James Bell's work on foods. It consisted in whisking the milk with a small brush made of fine brass wire. In his own hands it had worked extremely well, determinations of fat made in sour milks agreeing very well with those made on the same milks when fresh, even when the milk was badly curdled and the cream all separated out on the top.

Mr. Cassal said he could confirm what Mr. Richmond had just said as to the possibility of mixing decomposed milks and getting fairly accurate determinations of fat in them. He thought, however, that while this could undoubtedly be done satisfactorily when the weather had not been abnormally hot, yet in very hot summers, such as had been experienced within recent years, forms of decomposition would set in which practically broke up the milk altogether, and there was no longer any possibility of getting even an accurate determination of the fat under such circumstances. He did not know whether the authors had carried out any experiments upon samples of milk which had been kept at abnormally high temperatures, but from his own experience he might mention that it was a matter well worthy of their attention. A point of interest in connection with this method, in view of the existing necessity for stating analytical details in certificates given under the Sale of Food and Drugs Act, upon samples of watered milk, was that there was nothing to prevent an analyst from forming his opinion as to the purity of a sample of milk from such a factor as the nitrogen. It was a matter of considerable interest to him to find that Mr. Richmond, Mr. Smetham, and Mr. Allen were now suggesting a limit of 0·5 per cent. of nitrogen, as he (Mr. Cassal) had had occasion to state in court that a public analyst was not bound to found his opinion upon the solids-not-fat figure, and that he might, for instance, base his opinion on the amount of nitrogen. He presumed that they would consider anything very much below 0·5 to be indicative of adultera-

tion. If satisfactory results could be obtained by using this figure, instead of making out his certificate in the form recommended by the Society of Public Analysts, a public analyst could state that the percentage of nitrogen was the basis on which he had formed his opinion. It would be interesting if the authors could give some further information with regard to the "Dutch cows" referred to in the paper. It was quite plain that a liquid drawn from a cow's udder was not necessarily milk, and he suggested that the "Dutch cow" was no better than the remarkable "single cow" whose curious products had been so often called attention to. Far more information, of a trustworthy kind, would have to be obtained before any importance could be attached to the "Dutch" and "single cow" results.

Mr. R. A. CRIPPS remarked that whenever the percentage of non-fatty solids was considered to be unduly low, it would appear desirable that the inferences drawn from this should be confirmed by a determination of the nitrogen.

Dr. RIDEAL remarked that, although the figures were conclusive as to the absence of any evolution of nitrogen under the conditions of these experiments, it was by no means certain that, under ordinary practical conditions, changes might not take place after some time, resulting in an appreciable loss of nitrogen.

The PRESIDENT said that before asking Mr. Smetham to reply he would like to mention the fact that many Dutch cows did contribute to the milk-supply of London. Dutch cows were sometimes kept by dairy farmers on account of their very copious yield of milk. They yielded poor milk, but a large quantity of it. Thanks, however, to the activity and watchfulness of companies and wholesale firms such as those for whom Mr. Richmond and Mr. Woosnam acted, a pretty sharp watch was kept upon these large milk producers, and if they kept a fair number of Dutch cows they had to keep a large number of shorthorns as well, in order to swamp the poor quality of the Dutch milk. During the two previous years of drought a good many samples of milk had been sent to him by indignant vendors, who had received unpleasant letters from the wholesale dealers whom they supplied, on account of their milk coming dangerously near to or just passing the limits prescribed by the Society of Public Analysts. But in years of drought there was no doubt a tendency for all milk to be rather poor, and then the poverty of the Dutch cows very often made its effect felt, despite the mixture of the milk with that of shorthorns. He did not think many people would keep Dutch cows only, or that, if a man kept only a few cows, he would choose Dutch ones, as it would be too risky. What the Society of Public Analysts was hoping for was to obtain a legal definition of milk, or, rather, to get powers given to that Reference Committee which it was hoped would be legally constituted, to declare from time to time a definition of milk which would have the force of law. Then, no doubt, if it were decided that when a purchaser asked for "milk" he was entitled to get something containing 8·5 per cent. of non-fatty solids and so much fat, the vendor would use Dutch cows at his own risk and peril. A magistrate would probably take a lenient view in such a case if the man could prove the genuineness of his milk; but what they hoped was that they would get, through legal machinery, some such definition as would at any rate leave the *onus probandi* on the vendor in the case of anything falling below the limit fixed. Phenolphthalein always seemed to him to be a misleading indicator to use for determining acidity in a fluid

like milk, determining, as it did, the carbonic acid, which did not represent acidity in the sense of the term as used in reference to milk.

Mr. SMETHAM said that, although Dutch cows appeared to be used to some extent in the South, they had not, except in a few instances, penetrated to any large extent into the North. Lord Egerton, however, had on his estate a considerable herd, and it was from this herd that he (Mr. Smetham) had obtained all the samples of milk from Dutch cows which they had examined. It was, perhaps, unfortunate, but he figured out from the cost of feeding, the production of milk, etc., in the case of the last four Dutch cows, as compared with shorthorns and Jerseys fed at the same time, that the Dutch cows were the most profitable. The quantity of butter yielded per day by the shorthorns was 4·05 lb.; the Dutch cows gave 4·33 lb., the Kerrys 3·96 lb., and the Jerseys 4·50 lb. Of casein and albumin together the shorthorns gave 3·66 lb. per day, the Dutch 3·80 lb., the Kerrys 2·63 lb., and the Jerseys 2·87 lb. The Jerseys were, therefore, the most profitable from a butter-making point of view, but for cheese-making the Dutch were undoubtedly the most valuable, giving much more casein than any of the others. From the point of view of the farmer, whatever they might be from the consumer's point of view, the Dutch were undoubtedly the most economical cows to keep. With regard to the variations that had been alluded to, it had been pointed out in the paper that any calculations based upon the nitrogen factor were not as accurate throughout the whole of these tests as those based on the total solids-not-fat. Of course, the percentage of solids-not-fat recorded in the case of the Dutch cows would in itself represent about 7 per cent. of adulteration; if the percentage of solids-not-fat had been normal, no doubt the nitrogen would have been higher. He might mention that on several previous occasions he had examined milk from Dutch cows, and had always found it exceedingly poor, generally containing less than 8·5 per cent. of non-fatty solids. His experience, however, had been limited entirely to Lord Egerton's estate, and possibly there might be something in the climate or soil of that part of the country which gave rise to these abnormal results. So far as feeding was concerned, these cows were treated in exactly the same way as the Jerseys, which gave 9½ per cent. of solids-not-fat. They had used phenolphthalein as an indicator because of its very easy working and the very accurate results that it gave, but the figures for acidity stated in the paper represented the total acidity, from whatever source, and he did not for a moment wish to assume that it was all due to lactic acid. It was very possible, though he was not prepared to say that it was so, that the explanation suggested by Mr. Richmond for the loss of acidity in the milk after boiling was the correct one. He himself, however, rather preferred to look upon it as due to the presence of carbonic acid; but he had made no experiments to prove this, and only knew that they obtained in the milk as it came from the cow a result higher than that given by the same milk after it had been carried from the cow and mixed in a vat. The next day they took some milk direct from the cow and boiled it, and found that there was the dimunition in acidity which they had referred to, and which he attributed, rightly or wrongly, to loss of carbonic acid. With regard to Mr. Woosnam's remarks, he desired to point out that, although these samples were carefully weighed out while the milk was fresh, any difficulties

caused by bad condition of samples would have to be dealt with, whatever might be the mode of analysis. Difficulties from this cause were inherent to the analysis of a decomposed milk, but he thought the nitrogen determination would probably be affected in a less degree than any other. It would scarcely be possible to take 20 grammes for the determination of the total solids or the ash, but this quantity could be taken for the nitrogen determination, and was, in fact, about the best quantity to work on. This was a great advantage in favour of the Kjeldahl process. With regard to the temperature at which the experimental samples had been kept, there had been no attempt made to regulate it. The samples were in small bottles closed by corks, the bottles having been first sterilized. They were kept for a period extending from December, 1896, until the end of April, 1897, when some of these determinations were made, and although there had been no hot weather, some of them had occasionally been subjected to a rather high temperature, for at times the laboratory temperature rose as high as 70° F. So far as the experiments had gone, there was no appreciable loss of nitrogen, but they had been careful not to say that there could not be loss of nitrogen under conditions other than those under which the experiments were carried out. The Worleston milk was from an experimental farm belonging to the Cheshire County Council, and the feeding of the cows was as stated in the paper. He had for two years carried on experiments on this farm, and there was no doubt that very often, from one cause or another, the total solids of this milk fell to 8·5 and even lower. No doubt, as Mr. Bevan had said, the ash was a very constant constituent of milk, and a very good indication of its purity or otherwise, but it needed to be determined with considerable care. It was not an uncommon thing for inexperienced operators to get results that were too low, owing to volatilization of alkaline salts resulting from the use of too intense a flame. There was also the objection that if boric acid or a borate were present, the calculation was upset. He had made some experiments with regard to the percentage of phosphates in the ash of milk, and had found that phosphates formed a very constant constituent. The advantage, however, which the nitrogen had over the ash was that, except in the shape of ammonia salts, there was no very easy way in which a milkman could add any nitrogenous compound to the milk, and few would be likely to know how to add ammonia salts, or where to buy them.

NOTES ON THE INFLUENCE OF BORIC ACID UPON THE ACTION OF THE DIGESTIVE FERMENTS.

By R. A. Cripps, F.I.C.

(Read at the Meeting, May 5, 1897.)

The widespread employment of boric acid as a preservative of milk, fish, and other articles of food renders the subject of this short paper of the greatest importance.

It seems probable that the few simple experiments herein recorded have been long ago carried out on behalf of those whose commercial interests are largely influenced by the use of preservatives for food, but the fact that the results of any

such experiments have not, so far as I am aware, been hitherto recorded, must serve as my excuse for bringing so simple a matter before you this evening.

For the purposes of experiment the following were employed:

1. *Amylolytic ferment*—(*a*) as infusion of malt, (*b*) as saliva.
2. *Proteolytic ferment*—(*a*) as pepsin, (*b*) as zymin.
3. *Milk-curdling ferment* as chymosin.

I. Amylolytic Ferment.

(*a*) *Infusion of Malt.*—This was applied by a modification of the method of determining the diastatic power of extract of malt, described by me in the *Pharm. Journal*, 3rd Series, xx., p. 481. For this process three solutions are required: (1) Perfectly smooth potato-starch mucilage 1 per cent. (2) Malt liquor, prepared by digesting 1 part of good ground malt with 10 parts of distilled water at a temperature of 50° to 55° C. for an hour, and filtering. (3) One per cent. solution of iodine in KI.

Solutions 1 and 2 being raised to 38° C., 50 c.c. of 1 was introduced into each of a series of bottles, together with 50 c.c. of distilled water, containing the following quantities of boric acid: None, 0·01, 0·02, 0·05, 0·1, 0·2, 0·5, and 1·0 gramme. When the temperature was properly adjusted 5 c.c. of the malt solution was added to each, the bottles replaced in the bath, and small portions of each tested with iodine solution after five, seven, ten, and twelve minutes. In every instance digestion was complete at the end of twelve minutes, although not quite completed in ten minutes.

(*b*) *Saliva.*—One part by volume of fresh saliva was mixed with 9 parts of distilled water and strained. Into each of a series of bottles was introduced 10 c.c. of starch solution, with 12 c.c. distilled water containing boric acid in proportion to yield 1·0, 0·5, 0·2, 0·1, 0·05, 0·02, 0·01, and 0·0 per cent. of the final volume, raised to 38° C., and 2 c.c. of saliva solution added.

These were digested on the water-bath at a temperature of 38° C., the liquors being tested with iodine in the ordinary way. After four minutes all showed the red dextrin reaction, and all starch was *completely* converted in the following times:

0·2 per cent. boric acid after eight and a half minutes.
0·5 per cent. and 1·0 per cent. boric acid after nine minutes.
0·01 per cent. and ·05 per cent. boric acid after ten minutes.
0·02 per cent. boric acid after ten and a half minutes.
None and 0·10 per cent. boric acid after eleven and a half minutes.

II. Proteolytic Ferment.

(*a*) *Pepsin.*—For each experiment 5 grammes of white of egg in a fine state of division were mixed with 100 c.c. of water acidulated with 1 per cent. (vol.) of hydrochloric acid of officinal strength, the whole raised to 38° C., and the following proportions of boric acid added together with 5 c.c. of a 2 per cent. solution of pepsin—boric acid: (1) none, (1*a*) none, (2) 1 per cent., (3) 0·5 per cent., (4) 0·2 per cent., (5 and 5*a*) 0·1 per cent., (6 and 6*a*) 0·05 per cent., (7 and 7*a*) 0·02 per cent., (and 8 8*a*) 0·01 per cent. Digestion of Nos. 2, 3, and 7 was complete in forty-five minutes; Nos. 1, 4, 5, 5*a*, 6, and 8*a* in fifty minutes; and of Nos. 1*a*, 6*a*, 7*a*, and 8 in one hour. As is usual in such experiments, those in which digestion was most retarded showed

practically complete digestion by the time the first was completed. The tests, however, indicate that even 1 per cent. of boric acid does not retard peptic digestion.

(*b*) *Zymin.*—The proteolytic action of this ferment was determined by a modification of the U.S.P. process for pancreatine as follows:

(1) A solution was prepared containing 0·25 per cent. zymine and 0·75 per cent. sodium bicarbonate.

(2) A 1 per cent. solution of boric acid in fresh milk.

Solution No. 2 was mixed with such proportions of fresh milk as to produce 1·0, 0·5, 0·2, 0·1, 0·05, 0·02, and 0·01 per cent. boric solution.

These, and also a blank containing milk alone, were raised to 42° C., 25 c.c. of the zymine solution added and maintained at 35° to 38° C.

To test for completion of peptonization, to 1 c.c. of nitric acid of 2 per cent. by vol. were added 5 c.c. of the mixture. After thirty minutes, digestion in none of the liquids was completed, 20 c.c. more zymine solution was added, and the tests continued at intervals of about ten to fifteen minutes. After two hours twenty minutes all except that containing 1 per cent. boric acid no longer curdled when tested, and after two and a half hours even this was also digested.

III. Milk-curdling Ferment.

For this test 100 c.c. of fresh milk at 38° C. were rapidly mixed with 1 c.c. of a 2 per cent. solution of Blumenthal's chymosin and set aside. Similar experiments were made with milk containing from 0·01 to 1·0 per cent. of boric acid as before. In every instance a fine curd was produced in from six to seven minutes, that containing 1 per cent. of boric acid being certainly the firmest.

The practical result of these tests is that in so far as the processes of digestion in a living frame are represented by such experiments in glass vessels, such processes are not influenced by the employment of boric acid in quantity considerably in excess of that necessary for preservation of food; the question of its employment for such purposes would seem rather to turn upon the results of an inquiry into the physiological action of the acid when absorbed into the blood.

Pending such an investigation, it appears to me that the use of boric acid in reasonable proportion can scarcely with justice be made the ground of prosecution under the Food and Drugs Act.

Discussion.

The President said it was interesting to note that these results agreed closely, at any rate in the matter of peptic digestion, with the results of some similar experiments carried out by Mr. Hehner six or seven years previously. Mr. Hehner had taken white of egg, and pepsin, and dilute hydrochloric acid made up to represent gastric juice, and added varying quantities of boric acid up to about 1 per cent. of the whole solution. The conclusion Mr. Hehner came to was that peptic digestion was not in any way retarded by the boric acid. He pointed out, however, as Mr. Cripps had done, that this did not necessarily carry with it the inference that boric acid was harmless, for it was not known what it might or might not do when absorbed into the system. Some work had been done, he believed, in

America, previously to Mr. Hehner's experiments, with regard to the possible interference of boric acid with diastatic fermentation, and it was also then found not to have any retarding effect.

Mr. RICHMOND said that the experiments referred to by the President as having been made in America were probably those of Messrs. Leffmann and Beam, the results of which were published in the ANALYST nearly ten years ago. He would like to ask Mr. Cripps if the zymine which he used was that which was known as Fairchild's preparation. He (Mr. Richmond) was under the impression that Fairchild's preparation was supplied, mixed with the requisite quantity of alkali, and if Mr. Cripps had added alkali to this the solution would probably be too alkaline. He did not know whether, if this were the case, it would have any appreciable effect on the action of the boric acid.

Dr. RIDEAL said he thought he was correct in saying that, in addition to Messrs. Leffmann and Beam's experiments, some investigations had been made by H. A. Weber (*Journ. Amer. Chem. Soc.*, 1892, p. 4) as to the influence of boric acid on peptic digestion; and he had himself published in the *Pharmaceutical Journal* ("Year-book of Pharmacy," 1894, p. 443), the results of some experiments on papain digestion, in the presence of different acids, and found that boric acid made the least variation in the amount of digestion. He thought that, although boric acid might have no retarding influence upon normal digestion, its physiological action, as Mr. Cripps had mentioned, might be more marked. What public analysts, however, were concerned with was whether or not it was a "foreign" substance, and not whether it did or did not retard digestion. He considered that one very important point in connection with all artificial digestive experiments was that it was not correct to regard the disappearance of the substance experimented upon as the measure of the digestive action. That the starch or albumin dissolved by no means proved true digestion to have taken place. The starch might be converted into soluble starch, or the albumin into albumose, neither of which reactions constituted true digestion. The measure of the digestion of albumin was its conversion into peptone, and the measure of the digestion of starch was its conversion into dextrin, or, possibly, sugar.

Mr. S. F. BURFORD said that in some towns it was the practice for milkmen to take out milk and leave it in small shops during the whole day, collecting what remained unsold in the evening. He had met with more than one sample of milk that had been exposed in this way, and although the milk was chemically unobjectionable, its odour and taste were very bad indeed. It was scarcely necessary to remark that it would not be possible to leave milk exposed in this manner without any change being apparent, unless boric acid or some other preservative had been added to it; and he thought that this alone was a sufficient argument against the use of such substances.

Mr. CASSAL said that boric acid and borax were foreign substances, and therefore adulterants. They ought not to be present in articles of food unless their presence was acknowledged. If they were present, the purchaser had a right to be made acquainted with the fact. The mere fact that a preservative like boric acid was present in the food in sufficient quantity to produce its specific effect in retard-

ing or preventing the decomposition of that food was enough to show that its presence must affect the process of digestion. If such experiments as those described could be taken to represent what went on in the animal body, the conclusions drawn from them by the author might be justifiable; but such experiments could not be taken as representing the actual processes going on in the body in any way, and he presumed that Mr. Cripps would not seriously contend that they could. There was distinct evidence, on the other hand, that when boric acid was introduced into the animal body it produced injurious effects. A case had recently occurred in the neighbourhood of Dover, in which it was shown apparently beyond all doubt that fowls had been injuriously affected, and that some children had also been injured, by the ingestion of food containing boric acid. The food was a pudding made with milk containing rather a larger quantity of boric acid than was generally present, owing to the addition of the preservative by both the wholesale and retail vendors. Evidence had also been adduced which showed that boric acid and borax injuriously affected the kidneys; it had been recently used in the treatment of certain nervous diseases. The late Sir Andrew Clark, Sir Henry Thompson, and other well-known medical authorities had expressed themselves strongly on the subject. From the point of view of the public analyst, the presence of a foreign substance in a food constituted adulteration, and when the physiological effects produced by the ingestion of that foreign substance were even only doubtful, an offence of a serious character was constituted in the absence of any acknowledgment of the admixture. He regarded the addition of boric acid preservatives to milk, at any rate, as a grave offence indeed when the fact of the admixture was not disclosed, and nothing that the author had said could excuse or palliate that offence. The unacknowledged addition of so-called "preservatives," "sweeteners," and "antiseptics" to articles of food was largely on the increase, and it was a serious public evil.

The PRESIDENT observed that it was desirable to know whether the actual production of pepsin might not be retarded, even in the absence of any interference with its action in the stomach.

Mr. CRIPPS said that he had not been aware of the papers by Messrs. Leffmann and Beam and by Mr. Hehner, but was pleased to know that his own results were in accord with theirs. In reply to Mr. Richmond's question, he might say that he had used Fairchild's preparation of zymine as it was sold to him (containing one part of zymine to three parts of sodium bicarbonate), without any further addition of alkali. His own opinion was that the disappearance of the starch or albumin gave a very good indication of digestive power. He had, in fact, in the case of the experiments with starch, tested up to the point of complete digestion, namely, until iodine gave no more colour. There were certainly difficulties in the case of albumin, but he thought decidedly that the time taken to produce a given result was a very fair indication indeed of digestive power. A great many experiments in connection with the subject of peptic digestion had been published which confirmed this view. He could not agree with the opinion held by some that because boric acid was a substance foreign to milk, its use should be entirely prohibited. It was exceedingly difficult to keep milk, and he ventured to think that there existed practically a necessity for some preservative, provided that the use of such preservative could be shown to be harm-

less. Boric acid was in use to an enormous extent, and in this way had stood the test of experience for several years; if it was used in reasonable quantity he did not think any case could be brought forward proving it to be harmful. Its possible effect upon the production of pepsin, to which the President had alluded, was a question entirely for the physiologists, and one which he (Mr. Cripps) was quite unable to deal with.

The PRESIDENT said that, having regard to the present state of the physiological problem, he did not think it could be said that the addition of boric acid was an adulteration unless it was undeclared; but if its presence were undeclared, since boric acid was undoubtedly a foreign substance, the case was different, for anything sold as milk which contained an added foreign substance ceased to be milk, even irrespectively of the possibly injurious nature of the article added. Whether it should be considered illegitimate to sell milk containing boric acid, even with a declaration, on the ground that it contained a substance injurious to health, was a problem that undoubtedly was not yet solved, although the fact that, in small quantities, boric acid could not be very injurious to ordinary adult members of the population might almost be said to have been proved statistically, since we nowadays consumed so much of it in bacon, sausages, herrings, butter, and other substances besides milk. In the case of milk, however, the question might assume a very serious aspect, because infants were fed on milk, and it was possible that boric acid might have some injurious effect upon infants. Until this part of the question was satisfactorily settled by physiologists, he did not think that the unrestricted use of boric acid ought to be sanctioned. If it were made absolutely necessary to declare its presence, the public could then use their own discretion as to whether or not they would buy milk containing it, and they would have the option of getting milk free from preservatives for use in feeding infants. Whatever individual opinions were held in connection with this subject, he was sure it would be agreed that thanks were due to Mr. Cripps for his paper. Although some of the work might not be new, it was none the less interesting on that account, for confirmatory work by independent observers was always valuable.

NOTE ON THE PRESENCE OF HEAVY METALS IN CHEESE.

BY ALFRED H. ALLEN AND F. HUDSON COX.

(Read at the Meeting, June 2, 1897.)

IT will be within the recollection of those present that a paper was recently read before the Society by Mr. F. W. Stoddart, describing a sample of Canadian cheese in which he found metallic lead. In the discussion of that paper one of us made the statement that it was within his knowledge that sulphate of zinc was sometimes added to cheese. As this fact appeared to be outside the experience of most of the members of the Society, we have been at some trouble to ascertain how far the practice is still extant.

With this object we wrote to druggists in business in various parts of Wiltshire, asking to be supplied with samples of "Cheese Spice," that being the name by which

the preparation is locally known. In due course we received two samples of so-called "Cheese Spice," but other druggists to whom we applied stated that they did not sell it. Examination of these two samples showed one to be crystallized sulphate of zinc, while the other consisted of a solution of sulphate of zinc of such a strength as would correspond to 38 per cent. of the crystallized salt.

It appears that the "Cheese Spice" is used to prevent the heaving and cracking of cheese.

The question next suggested itself whether the zinc used remained in the curd or passed away in solution in the whey, and to settle this point we prepared a small quantity of cheese by means of rennet, adding sulphate of zinc in the proportion of 1 gramme to the gallon of milk, which is about that in which it is directed to be used. As a consequence we find that 99·28 per cent. of the total zinc employed passed into the curd, the remaining 0·72 per cent. remaining in solution.

Mr. R. Bodmer informs us that he quite recently found zinc in two specimens of cheese sold in Southwark, which were submitted to him under the Sale of Food and Drugs Act. One of these was a pale-coloured cheese, similar in appearance to Cheddar, and contained 0·84 grain per lb. of zinc, corresponding to 3·7 grains per lb. of the crystallized sulphate. The other sample was a reddish cheese, more like Gloucester, and contained 0·56 grain per lb. of zinc, corresponding to 2·5 grains of the crystallized sulphate.

From these facts there is no doubt that the practice of adding sulphate of zinc to cheese is not obsolete, although it is apparently dying. There can be little doubt that the zinc in the samples analysed by Mr. Bodmer owed its origin to the practice we have described, and we would suggest that the metallic lead found by Mr. Stoddart had its origin in the addition of some preparation of lead, probably the acetate, to the cheese.

It is within our knowledge that sulphate of copper was formerly added to cheese, and it is well-known that the green mould in certain kinds of cheese has been imitated by the insertion of copper or brass skewers.

Instances are on record of chromate of lead having been found on the outside of cheese, its presence being attributed to the cheese having been wrapped in a yellow cloth.

Mr. A. Wynter Blyth, in his work on "Foods," states that in past times instances have occurred in which preparations of arsenic have been added to cheese as a preservative, and that in 1841 several persons were poisoned by these means. A similar case occurred in 1854, when a Parisian family suffered, but not fatally.

Discussion.

The President said it seemed very curious that a substance of this kind should be used at all. There was not the slightest necessity for the use of any kind of antiseptic in cheese-making except salt. At the best it could only be required as a corrective for very slovenly dairy work. It was unfortunate that Dr. Stevenson was not present, for he had at one time paid a good deal of attention to the toxic action of small quantities of zinc.

Mr. BEVAN remarked that the bottle containing the solution bore no "poison" label.

Mr. ALLEN said that zinc was not in the poison schedule, hence such a label was not legally required.

Mr. HARVEY thought that zinc would act as an emetic rather than as a poison.

Mr. BODMER said that this was on a line with the question of copper in peas. Continued small doses would probably have a much more serious effect than a single large dose.

Mr. HARVEY remarked that copper was a cumulative poison, whereas zinc was not.

Mr. BODMER said that in the matter of toxic action zinc was by some authorities ranked higher than copper, while by others it was regarded as lower than copper.

ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

FOOD AND DRUGS ANALYSIS.

Critical Examination of the Iodine Number of Lard. W. Meyer. (*Zeit. für öffent. Chemie*, iii., 134.)—Samples of lard examined during the past two years gave 65·0 and 54·9 as the highest and lowest iodine numbers, whilst the average of thirty-four samples was 59·4. The samples giving iodine numbers above 60 came through Hamburg houses, and were probably of American origin, whilst those with the lower iodine numbers were from small home slaughterers. In none of the samples could admixture of foreign fat be detected.

A recently-examined sample gave the low iodine number 49·7, and although, according to Späth and Neufeld, so low a number is possible, the sample was further tested for cocoa-nut-oil and beef-fat—the only adulterants ever used—but no evidence of either could be obtained.

Two other samples from reliable sources gave 50·8 and 52·1.

In three cases, in which the lard was prepared in the laboratory from different descriptions of pig-fat, the iodine numbers were 47·5, 49·8, and 58·4.

The author agrees with Mansfeld in fixing the upper limit at 65·5, and with Neufeld in placing the lower at 46·0. H. H. B. S.

Determination of Caffeine in Coffee. A. Forster and R. Riechelmann. (*Zeit. für öffent. Chemie*, iii., 129.)—Twenty grammes of ground roasted coffee are boiled four times with 200 c.c. of water, the extract made up to a litre, filtered, and 600 c.c. of the filtrate placed in an extraction apparatus with some chloroform. Solution of sodium hydrate is then added to alkaline reaction, and the whole digested with chloroform for ten hours. The chloroform extract is now transferred to a flask, the chloroform distilled off, and the nitrogen determined in the residue by Kjeldahl's

method, the caffeine being calculated from the nitrogen found. A sketch is given in the original paper of the apparatus which the authors used for the extraction.

In the course of their experiments, the authors found in aqueous coffee extract a body which they believe to be identical with the new alkaloid which has been named "coffearine" by Paladino. The new alkaloid is precipitated by picric acid, is insoluble in chloroform, and fails to give the murexide reaction, by which characteristics it is distinguished from caffeine.

In order to ascertain the relative proportions of the two alkaloids in coffee, the authors determined the total alkaloid nitrogen and the caffeine nitrogen, assuming the difference between the two to represent the nitrogen existing as coffearine. In this way they obtained the following results with three different sorts of coffee:

Nitrogen as Coffearine.	Nitrogen as Caffeine.
0·154	0·344
0·203	0·320
0·043	0·370

H. H. B. S.

On the Determination of Caffeine in Coffee and Tea. A. Hilger and A. Juckenack. (*Forsch. Ber.*, 1897, iv., 49, 50.)—Twenty grammes of the finely-powdered tea or coffee (previously dried in the water-oven in the case of unroasted coffee) are extracted with 900 c.c. of water for several hours at the ordinary temperature, and then completely exhausted by boiling with water. The extract is cooled to about 60 to 80°, 75 c.c. of a solution of basic aluminium acetate (liquor aluminii acetici of the German Pharmacopœia) added, and then by degrees with continual stirring 1·9 grammes of sodium bicarbonate. After being boiled for five minutes, the liquid is cooled, brought to a total weight of 1,020 grammes, and filtered. Seven hundred and fifty grammes of the clear filtrate (corresponding to 15 grammes of the original substance) are mixed with 10 grammes of precipitated aluminium hydrate and some paper pulp and evaporated to dryness. The residue is well dried in the water-oven and extracted for eight hours with carbon tetrachloride in a Soxhlet apparatus. The solvent is evaporated and the residue of caffeine, which is quite colorless, is dried and weighed.

Where absolute accuracy is required, the nitrogen in the crude caffeine is determined by Kjeldahl's method, and the result calculated to water-free caffeine, 1 c.c. of $\frac{N}{10}$ sulphuric acid corresponding to 0·00485 gramme of caffeine. The results obtained in this way are 2 to 4 milligrammes less than those yielded directly. The carbon tetrachloride requires to be purified by shaking with 5 per cent. soda solution, washing with water and drying with calcium chloride before it can be used for the extraction. C. A. M.

On the Relation between the Amount of Caffeine and Quality of Chinese Tea. L. Graf. (*Forsch. Ber.*, 1897, iv., 88, 89.)—Kellner has shown that in the living tea-plant the amount of caffeine in the leaves decreases with the growth.

Since the finer sorts of tea consist of young leaves, it occurred to the author that the percentage of caffeine might serve as an indication of the quality. Two varieties of tea were examined, and the caffeine was estimated by making an aqueous extract, precipitating the tannin with basic lead acetate, filtering, removing the lead, concentrating the filtrate after the addition of sodium acetate, again filtering and extracting the caffeine with chloroform.

The results calculated on the air-dried tea were :

	No.	Caffeine, per cent.	Price, Marks.
Souchong	206	2·96	1·30
,, ...	273	3·10	1·80
,, ...	266	3·53	3·15
Congou	270	2·82	1·80
,, ...	269	3·70	2·40
,, ...	268	4·09	3·10

Thus in both kinds of tea the amount of caffeine increases with the quality and commercial value. C. A. M.

Estimation of Theobromine in Cocoa and Chocolate. L. Maupy. (*Journ. Pharm. Chim.*, 1897, v., 329-332.)—Five grammes of cocoa, from which the fat has been extracted with petroleum spirit, are triturated with 2 c.c. of water and then boiled under a reflux condenser for an hour with a mixture of 15 grammes of phenol and 85 grammes of chloroform. When cool the phenol-chloroform extract is filtered, and the residue twice decocted for thirty minutes with 15 grammes of chloroform, the decoctions being added to the filtered solution. The chloroform is evaporated, and 40 grammes of ether are added to the residue and left for six hours. The theobromine is precipitated, while the caffeine, colouring matter and last traces of fat remain in solution. The precipitate is collected on a weighed filter, washed with several c.c. of ether to remove phenol, dried, and weighed.

In the case of chocolate, 10 grammes are finely powdered, and the fat extracted with petroleum spirit. The residue is mixed with 4 grammes of 70 per cent. alcohol, and the estimation continued as if one were working on the 5 grammes of cocoa. When the proportion of sugar in the chocolate is more than 50 per cent. the quantity of phenol in the solvent is decreased, so that about 0·6 gramme of phenol are used for each 1 gramme of cocoa.

The following results were obtained in this way with samples of roasted cocoa :

	Theobromine, per cent.
Cocoa Trinidad	1·44
,, Caracas	1·38
,, Para	1·28
,, Grenada	1·60
,, Martinique	1·52

A chocolate containing 60 per cent. of sugar yielded 0·54 per cent. of theobromine.

It is stated that the alkaloid is white or only slightly colored, even in the case of roasted cocoa and chocolate.

C. A. M.

Analysis and Composition of Liquorice Extract. G. Py. (*Journ. Pharm. Chim.*, 1897, 280-284.)—*Moisture.*—Ten grammes of the liquorice extract are dried for seven hours at 100°.

Ash.—The residue from the above is used for the estimation of the soluble and insoluble ash.

Alcoholic Extract.—This is found by dissolving 2 grammes of the extract in about 30 c.c. of water, cooling, and adding alcohol so as to make the mixture contain 75 per cent. After being stirred, the precipitate is allowed to settle for twelve hours, then collected on a filter and washed with 75 per cent. alcohol. The filtrate is evaporated, and the residue dried at 100° C. and weighed.

Ammonium Glycyrrhizate.—The alcoholic extract from the previous determination is dissolved in hot water and precipitated with sulphuric acid (1 : 10). The precipitate, after having been washed with water acidulated with sulphuric acid and then with distilled water, is dissolved off the filter by concentrated ammonia. The ammoniacal solution is evaporated, and the residue dried at 100° and weighed.

Organic Substances Precipitated by Alcohol are estimated by deducting from 100 the percentage of total ash, moisture, and alcoholic extract.

As a rule, these five determinations enable one to judge of the commercial value of a liquorice extract; but further information may be obtained by determining the nitrogen by Kjeldahl's method. The nitrogen belonging to the glycyrrhizin can be found by multiplying the amount of the latter by 0·0593.

Reducing Sugars are determined in the filtrate from the glycyrrhizin precipitate.

Detection of Gelatin.—Gelatin ought always to be looked for, since it is of frequent occurrence in liquorice extract, either fraudulently or to give suppleness to liquorice-sticks, and to prevent their sticking to the paper. It is tested for in the portion insoluble in 75 per cent. alcohol, which is dissolved in hot water and then cooled.

C. A. M.

ORGANIC ANALYSIS.

Some Colour Reactions of Tartaric, Citric, and Malic Acids. E. Pinerua. (*Ann. de Chimie Analyt.*, ii. [4], 66.)—The reagent employed is a freshly-prepared solution of 0·02 gramme of β-naphthol in 1 c.c. of sulphuric acid (specific gravity 1·83), from 10 to 15 drops being added to about 0·05 gramme of the acid under examination, the mixture being carefully and slowly warmed in a porcelain capsule.

Tartaric Acid.—Blue coloration, changing under the influence of heat to a decided green. When cooled and diluted with 15 to 20 volumes of water, the liquid becomes a persistent yellow-red.

Citric Acid.—Intense blue colour unchanged by a continuance of heat, but disappearing (or turning pale yellow) on dilution. The admixture of a small quantity of tartaric acid suffices to develop the characteristic green; a dull blue-green indicates 10 to 12 per mil. of this acid.

Malic Acid.—Greenish-yellow on progressive heating, turning to bright yellow, and becoming bright orange on dilution.

The reactions of the other organic acids are much less distinct than the above. In case of mixtures the acids should be separated before the test is applied. When the coloration begins to appear, the capsule should be withdrawn from the flame and only heated again in order to verify the change. C. S.

Research on Geranium-Oil. E. Charabot. (*Bull. Soc. Chim.*, 1897, xvii., 489-492.)—It has been shown that geranium-oil contains a mixture of two isomeric alcohols, geraniol and citronellol, which rotate the beam of polarized light in opposite directions. The author's experiments were made with the object of determining whether optically active esters were also present. For this purpose the samples were saponified, and the product distilled in a current of steam. In all cases there was a sensible diminution of the rotatory power. It was observed that the less volatile portion of the saponified essence had the disagreeable odour of the part passing over between 200° and 210°, when geranium-oil is fractionally distilled.

The results obtained with oils of known purity were as follow :

Origin.	Per Cent. Ester as Tiglate of Geranyl.	Free Alcohol as $C_{10}H_{18}O$.	Total Alcohol.	Specific Gravity at 15° C.	Rotatory Power l=100 m.	Rotatory Power after Saponification.	Lowering of Rotatory Power.
Algiers, 1893	25·31	46·22	62·74	0·896	–9° 50′	5° 46′	4° 4′
Algiers, 1895	22·11	50·80	65·23	0·899	–9° 20′	4° 24′	4° 56′
Algiers, 1896	23·32	60·30	75·52	0·898	9° 48′	5° 46′	4° 2′
Algiers, 1896	25·66	41·80	58·55	0·895	–10° 4′	–5°	5° 4′
Algiers, 1897	24·86	55·41	71·62	0·894	–9° 10′	5° 8′	4° 2′
Geranium Bourbon ...	32·16	46·12	67·11	0·8915	–9° 20′	–7° 40′	1° 40′

C. A. M.

INORGANIC ANALYSIS.

Analysis of Aluminium and its Alloys. F. Jean. (*Ann. de Chimie Analyt.*, ii. [4], 61-65.)—It is important to determine the proportions of metals and metalloids in aluminium, on account of their influence on the physical and mechanical properties of the metal; and the methods proposed by Moissan and Gouthière (ANALYST, vol. xxi., pp. 83 and 270), though complete, do not exhaust the subject.

The author acts cautiously on 10 grammes of metal with 10 per cent. hydrochloric acid, absorbing the evolved gases in a Will and Warrentrapp bulb-tube containing bromine; the solution thus obtained is afterwards heated to drive off the bromine, the *sulphur* thrown down as barium sulphate, and the *arsenic* precipitated by hydrogen sulphide. This latter precipitate is heated with fuming nitric acid, and so converted into arsenic acid, in which form it is weighed, any *antimony* present remaining undissolved as oxide, when the precipitate is again taken up with water. *Phosphoric acid* is determined from the filtrate by boiling, treating with 5 c.c. of nitric acid and 10 c.c. of concentrated ammonium nitrate, and precipitating with ammonium molybdate, the Pemberton method of titration being adopted.

The solution of the metal is diluted and filtered, *carbon* being determined from the loss on ignition of the residue dried at 100° C. The remainder of the residue is fused with potassium nitrate and alkali carbonates and taken up with boiling water, *silica* being rendered insoluble by evaporation to dryness with a little hydrochloric acid, whilst the other constituents are re-dissolved in nitro-hydrochloric acid and united to the main solution. From this solution *lead* is thrown down (in the absence of tin and antimony) by hydrogen sulphide, the precipitate being ignited, dissolved in nitric acid (with a few drops of sulphuric acid), and estimated as sulphate. *Copper* is precipitated from the filtrate by boiling with sodium hydroxide. Should *tin* or *antimony* be present, the sulphide precipitate is treated with yellow ammonium sulphide, and these metals estimated in the usual way.

A colorless filtrate from the hydrogen sulphide treatment indicates the absence of *chromium* and *nickel*, and in that event it is concentrated to 100 c.c., one half of which (= 5 grammes of metal) is treated with potassium chlorate, and evaporated to dryness to remove *silica*, which is fused with alkali carbonate and again separated to ensure absence of alumina. The filtrate is then treated with 25 to 30 c.c. of hydrochloric acid and titrated for iron with stannous chloride. *Manganese* and *zinc* are determined in the second 50 c.c. of solution. This is rendered alkaline by sodium hydroxide, boiled to dissolve the alumina, and treated with bromine to precipitate *manganese*, *zinc* being titrated with sodium sulphide.

When *chromium* is present, a separate portion of substance is taken for the estimation of this element and iron, 1 gramme being carefully attacked by sodium hydroxide, and afterwards fused with potassium nitrate and sodium bicarbonate, taken up again with water and filtered. The insoluble oxides are dissolved in dilute sulphuric acid, and the solution, after being reduced by zinc, is filtered off and titrated with permanganate for *iron*. *Chromium* is estimated in the solution colorimetrically, 50 c.c. being compared with an equal volume of standard solutions of neutral potassium chromate.

Nitrogen may be determined by passing through 10 c.c. of decinormal acid the gas resulting from the action of a solution of sodium hydroxide (S. G. 1·32) on 5 or 10 grammes of metal, distilling when the reaction is terminated, and titrating the ammonia by decinormal alkali.

The Moissan method of *sodium* determination may be advantageously modified by converting the nitrate into sulphate and then into carbonate (by calcination with ammonium carbonate), removing the sulphuric acid by baryta-water, and titrating this sodium salt (instead of the chloride) by decinormal acid. C. S.

The Separation of Iron from Aluminium. **F. A. Gooch and F. S. Havens.** (*Zeit. anorg. Chem.*, xiii., 6, 435-440.)—The method is based upon the solubility of ferric chloride in a mixture of equal parts of concentrated hydrochloric acid and anhydrous ether, aluminium chloride being practically insoluble in this medium. A number of results given show that the method is accurate, when carried out in the following way: The mixed chlorides are dissolved in a very small quantity of water; about 15 c.c. of a mixture of concentrated hydrochloric acid and ether are added,

and the whole cooled to 15° C. Hydrochloric acid gas is now passed in until the solution is quite saturated. Five c.c. more ether are added, and hydrochloric acid gas again passed in. The aluminium chloride is filtered off through an asbestos filter, washed with the above acid and ether mixture, which must be saturated with hydrochloric acid gas, and dried for half an hour at 150° C. The precipitate is then heated for some time with one gramme of mercuric oxide, and finally ignited over the blow-pipe. By this treatment the chloride is converted into oxide. An alternative method is to dissolve the precipitated aluminium chloride in dilute acid, and to precipitate with ammonia in the usual way. If much iron is present a green oily layer separates out on the addition of the acid and ether solution, consisting of an ethereal solution of ferric chloride. An excess of ether prevents the formation of this layer. W. P. S.

Determination of Nickel in Nickel-steel. J. Spüller. (*Chem. Zeit.*, xxi., 26, 243.)—A number of standard samples of nickel-steel filings are provided, in which the percentage of nickel is accurately known. For testing steels containing from 1 to 7 per cent. of nickel, it is sufficient to have three such standard samples with 1, 3, and 5 per cent. of nickel respectively. Two grammes of the sample to be tested, and 2 grammes of one or more of the standard samples, are each boiled with about 60 c.c. of nitric acid of 1·2 specific gravity in ¼-litre flasks until completely dissolved and fumes cease to be given off. After cooling, the iron is precipitated with zinc oxide, the flasks are made up to the mark, shaken, the precipitates allowed to settle, and the liquids filtered off, the green-colored filtrate from the sample under examination being compared with the corresponding filtrate from the standard samples.

H. H. B. S.

The Estimation of Vanadium by means of Organic Acids. Philip E. Browning and Richard J. Goodman. (*Zeit. anorg. Chem.*, xiii., 6, 427-434.)—On boiling a solution of ammonium vanadate with an excess of tartaric acid, the vanadic acid is reduced to the tetroxide, which may be titrated with a standard solution of iodine, after neutralization with sodium bicarbonate.

Tungstic acid is not reduced by tartaric acid, and its presence has no influence on the estimation of vanadic acid, whilst molybdic acid is readily reduced.

Experiments on the action of tartaric acid on vanadic acid in the cold proved that the reduction to the tetroxide was complete, provided that the solution was concentrated, and the time of action extended to one day, whilst under similar circumstances tungstic and molybdic acids showed no signs of reduction. Vanadic acid may therefore be estimated in the presence of both of these acids.

Oxalic or citric acid may be used in the place of tartaric acid. An excess of oxalic or tartaric acid has no influence on the estimation, but too large an amount of citric acid must not be used, as it tends to give somewhat high results.

The amount of organic acid used should be, approximately, ten times the amount of the vanadic acid to be estimated. It will be found better, in titrating the

tetroxide, to add a slight excess of the standard solution of iodine, and then to titrate this back with a standard solution of arsenious acid, using starch as an indicator. W. P. S.

The Reaction of Filter-paper. L. Magnier de la Source. (*Ann. de Chimie Analyt.*, ii. [5], 82-84.)—Having, contrary to the experience of R. Gans, found that on boiling tartar along with the filter-paper a slight error in excess always results, the author studied the reaction of several kinds of filter-paper, and found:

1. Ordinary filter-paper boiled in water colored rose by 1 drop of alcoholic phenolphthalein solution and 2 drops of lime-water, left the colour unaffected, but when decolorized by 1 c.c. of decinormal sulphuric acid, 0·0015 gramme of the latter was absorbed; this does not, however, prove combination of the acid with the cellulose of the paper, but may be merely due to the presence of earthy carbonates.

2. French "Berzelius" paper behaved in a perfectly neutral manner under the same treatment.

3. Schleicher and Schüll's "Berzelius" paper, washed with hydrochloric acid and hydrofluoric acid, immediately decolorized the indicator, and the restoration of the colour required an amount of lime-water equal to 0·0014 gramme of sulphuric acid.

4. Swedish "Berzelius" paper, over fifty years old, showed, before washing, a degree of acidity equivalent to 0·0003 gramme.

5. The same paper, washed with very dilute hydrochloric acid followed by distilled water till the washings were neutral, and dried in presence of potassium hydroxide, gave an acid reaction to the extent of 0·0008 gramme of sulphuric acid.

6. After the same paper had been subjected to prolonged washing with boiling water, its acidity declined to 0·0002 gramme, *i.e.*, about its initial degree.

The necessity for washing the filter with boiling water to remove the final traces of acidity, which would falsify the titration of alkali tartrate, is thus apparent, especially since even 0·0005 gramme of acidity will cause an error in excess of 0·2 gramme of tartrate in 10 c.c. C. S.

APPARATUS.

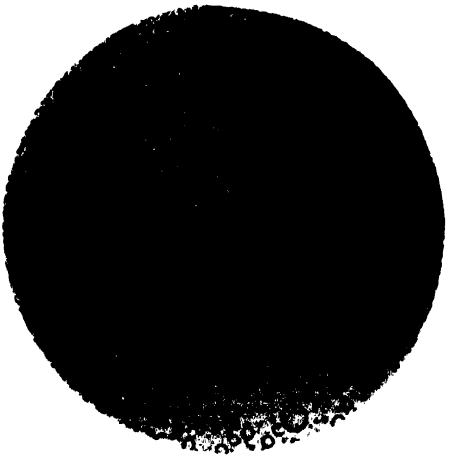
The Serrin Balance for Quick Weighings. A. Démichel. (*Rev. de Chim. Analyt.*, v. [2], 25-27.)—The distinguishing feature of this balance is that weights and riders of less than 1 decigramme are dispensed with, their place being taken by a chain, one end of which is suspended from one arm of the beam, and the other supported by a lever mounted in the case and capable of elevation or depression at will, whereby the effective weight of chain on the balance is adjusted. This weight is recorded by a graduated scale attached to the lever and sliding behind a vernier in one of the uprights of the case.

In weighing, the ordinary weights are employed to approximately balance the substance, the final adjustment being made by lowering the chain, and as this can be done without interruption or opening the case, the operation can be performed more rapidly than usual. The chain appliance can be fixed to any balance with a beam long enough to allow the chain to hang freely between the column and the pan.

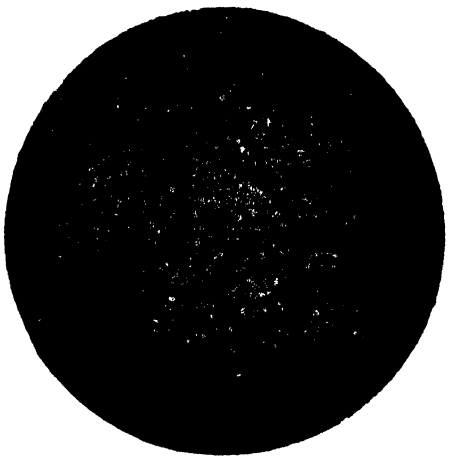
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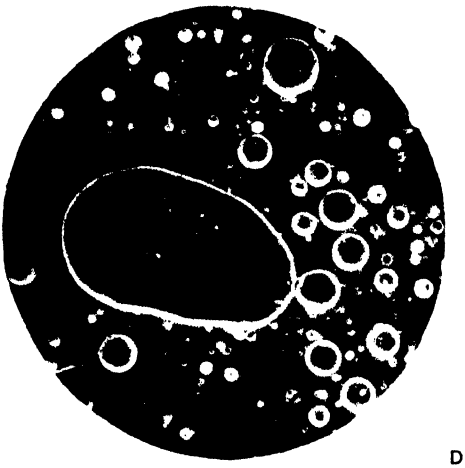
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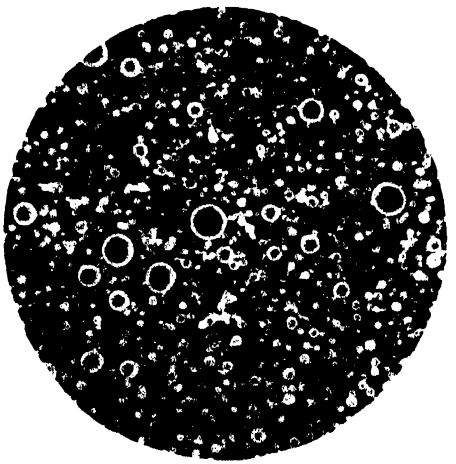
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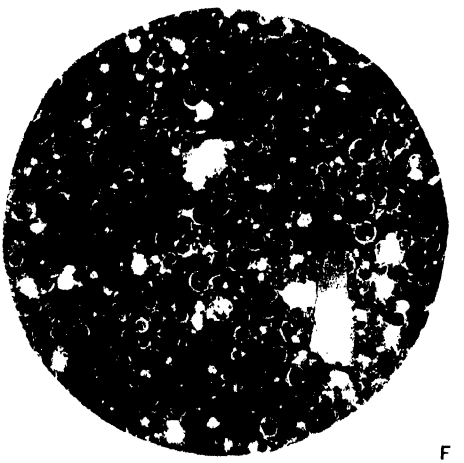
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THE ANALYST.

AUGUST, 1897.

OBITUARY.

Mr. F. M. RIMMINGTON.

We regret to announce the death, on June 17, of one of the oldest members of our Society, and one of the oldest Public Analysts in England, viz., Mr. F. M. Rimmington, Public Analyst for the boroughs of Bradford and Dewsbury. Mr. Rimmington was a pharmacist in Bradford for fifty years. He had always taken an active interest in the detection of adulteration of food and, perhaps more especially, of drugs; and on the passing of the Sale of Food and Drugs Act, he was appointed Public Analyst for the boroughs already mentioned, and retained those appointments until the time of his death, which occurred at the ripe old age of seventy-eight. He was the author of a number of papers, most of which have been published in the Journal of the Pharmaceutical Society, but some of which, on analytical subjects, have appeared from time to time in our own pages. Notwithstanding his advanced age, he still took an active interest in both analytical and general scientific work, and especially in microscopy. He was a man of striking personality, and will be missed, not only in his own town, but by a large number of friends and correspondents all over the country. Mr. F. W. Richardson, who was for many years Mr. Rimmington's assistant, has been appointed to succeed him as Public Analyst for Bradford.

B. D.

PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS.

ON THE STRUCTURE OF THE "FAT GLOBULES" IN COW'S MILK.

By Professor V. Storch, Copenhagen.

Translated and Communicated by Harald Faber.

(*Read at the Meeting, June* 2, 1897.)

From the analyses of the butter and butter-milk derived from the same churning the composition of the serum of the butter and the serum of the butter-milk can be calculated. By "serum" is understood the aqueous portion, *i.e.*, everything in the butter except fat and salt, and in the butter-milk everything except the fat. It will be found that the butter-serum always contains much more albuminoids and much

less milk-sugar than the serum of butter-milk. This will be seen from the following table, which shows the average composition of the butter and butter-milk from ten different churnings, the cream being churned both fresh and after ripening:

COMPOSITION OF BUTTER AND BUTTER-MILK.

	From *fresh* cream.		From *ripened* cream.	
	Butter.	Butter-milk.	Butter.	Butter-milk.
Albuminoids	0·64	3·28	0·84	3·37
Milk-sugar	0·35	4·98	0·39	4·58
Ash	0·14	0·79	0·16	0·81
Water	13·03	89·74	13·78	90·93
Fat	83·75	1·21	82·97	0·31
Salt	2·09	—	1·86	—
	100·00	100·00	100·00	100·00

From these figures the composition of the butter-serum and the butter-milk-serum can be calculated, as is shown in the following table:

COMPOSITION OF SERUM OF BUTTER AND BUTTER-MILK.

	From *fresh* cream.		From *ripened* cream.	
	Butter.	Butter-milk.	Butter.	Butter-milk.
Albuminoids	4·52	3·32	5·54	3·38
Milk-sugar	2·47	5·04	2·57	4·60
Ash	0·99	0·80	1·05	0·81
Water	92·02	90·84	90·84	91·21
	100·00	100·00	100·00	100·00

It will be seen that the serum of butter made from fresh cream contained 1·20 per cent. more albuminoids and 2·57 per cent. less milk-sugar than the serum of the butter-milk, and somewhat similar figures were found for the products derived from ripened cream. It is evident that the serum of butter cannot contain more of the serum derived from butter-milk than would correspond to its content of milk-sugar. In the serum of butter from fresh cream there can only be $\frac{2{\cdot}47}{5{\cdot}04} \times 100 = 49{\cdot}01$ per cent. of serum of butter-milk; and similarly that of butter from ripened cream can only contain 55·87 per cent. of the serum of butter-milk. If from the serum of butter this calculated amount of the serum of butter-milk is deducted, there will remain a *residue* consisting of albuminoids, ash, and water, which will have the following composition:

COMPOSITION OF THE RESIDUE FOUND BY CALCULATION TO BE PRESENT IN

	Butter from *fresh* cream.	Butter from *ripened* cream.
Albuminoids	5·67	8·27
Ash	1·18	1·36
Water	93·15	90·37
	100·00	100·00

This residue was found to be of very similar composition in all the ten churnings

where the cream had been churned fresh; and in all the ten churnings with ripened cream it also had almost the same composition as the average given above, but, as will be seen, there was a difference in the composition of this residue according as to whether it was derived from fresh or from ripened cream. The proportion between fat and residue in the butter was found to be almost the same in both series of churnings, inasmuch as 100 parts of fat corresponded to 8·62 and 8·07 parts of residue from fresh and ripened cream respectively. This discovery, made several years ago, led me to conclude that a certain relation could be found between the fat and this residue, and that perhaps this aqueous albuminoid forming the residue was connected in some way with the fat globules in the milk.

I therefore made a careful investigation of the albuminoids in butter by the following method: From 1 to 2 lb. of butter were melted at a low temperature until the butter-fat had clarified. The total content of serum and salt separated out as a white viscous mass. The butter-fat was carefully decanted off, the serum rinsed twice with benzene, afterwards diluted with distilled water, and carefully mixed with one and a half times its volume of strong alcohol. A white flocculent precipitate formed, which contained nearly all of the albuminoids from the butter, together with some salt. This was separated by filtration, washed with 60 per cent. alcohol, and extracted with ether until all fat was removed. On drying in the air at the ordinary temperature, this precipitate was converted into a very fine white powder. After drying, it was extracted with distilled water and washed until the washings were free from salt, and then treated with a 0·2 per cent. solution of sodium hydrate. Under this treatment it swelled very much, and formed a stiff gelatinous mass which could take up a considerable amount of water without losing its gelatinous consistency. Stirred up in a large quantity of water, the voluminous gelatinous precipitate slowly settled; it could only be separated from the liquid by filtration with extreme difficulty. After being washed by decantation, brought on to a filter, and treated with strong alcohol, it did not lose its gelatinous consistency. By subsequent washing with ether and drying in the air it formed a fine, loose, hygroscopic powder of grayish-white colour having the following properties. It was insoluble in water, alcohol, and ether, almost insoluble in acetic acid and dilute mineral acids at the ordinary temperature. In very weak solutions of sodium hydrate or ammonia it swelled considerably without dissolving. It dissolved slowly and incompletely in a dilute solution of sodium hydrate, and was precipitated therefrom by an acid. Even in concentrated solutions of sodium or potassium hydrate it dissolved very slowly and incompletely at the ordinary temperature, somewhat more quickly on heating; it then formed a light, yellowish-brown spongy mass. When swollen in water it gave the reactions characteristic of albuminoids—Millon's reaction, that with iodine, and the biuret reaction. It contained a considerable quantity of sulphur, of which only a small quantity was removed by boiling with alkalies. Treated with warm dilute hydrochloric acid, a substance was formed which reduced Fehling's solution. The content of nitrogen of the ash-free albuminoid was found to be 14·2 per cent., consequently this albuminoid must be totally different from the casein and albumin in milk. As this substance has been found in butter made from absolutely fresh cream, it cannot have been formed from the casein in the

cream by an alteration brought about during the ripening process by the action of micro-organisms. The albuminoid in question differs from casein not only in its insolubility in weak alkaline solutions and in dilute acetic acid (both of which dissolve casein readily), but also in its chemical composition, and by the fact of its yielding, when heated with dilute mineral acids, a substance which reduces Fehling's solution. That the reduction of the copper solution could not be attributed to milk-sugar was proved by the entire absence of the colour reaction with α-naphthol before treatment with dilute hydrochloric acid. The only albuminoid proper which forms a reducing substance on treating with dilute acid is serum-globulin, but it forms considerably less thereof than the albuminoid in question. That group of albuminoids which possesses this property (the so-called "gluco-proteids"—mucin—and several nucleo-proteids) differ in chemical composition from the albuminoid in question, as will be shown hereafter. This albuminoid forms at least between 60 and 63 per cent. of the total albuminoid contained in butter.

The residue calculated above from the difference between the butter-serum and the butter-milk-serum is composed of this new albuminoid. From the composition of the butter-serum and of the residue it can be calculated that this albuminoid forms over 60 per cent. of the total albuminoid contents of the butter-serum, or the same amount as found by direct estimation.

It has been shown that more than one-half of the total quantity of the albuminoids of butter consists of a peculiar albuminoid differing in its chemical properties and its chemical composition from the albuminoids hitherto found in milk, viz., casein, lact-albumin, and globulin. As its proportion in butter is, on the average, 0·45 per cent. by weight, it should be present in the milk in sufficient quantity to be identified. As already mentioned, I expected to find it in some way connected with the fat globules, and therefore attempted to free these from milk-serum by washing. Fresh cream was diluted with four times its volume of water and left for the cream to rise, but to my surprise very little cream rose, even after two days' standing. Fresh cream was therefore mixed with four times its volume of a 33 per cent. solution of cane-sugar, and left in a large separatory funnel. After standing for a day a thick layer of cream had formed, and the sugar solution below it had an opalescent appearance almost like a mixture of skim-milk and water. The sugar solution was allowed to drain away, and the remaining cream was again mixed with four times its volume of a 33 per cent. solution of sugar. The cream rose quickly and completely, and the only slightly opalescent sugar solution could be run off after six hours' standing. The cream was washed four times in this way, and as the sugar solution from the fourth washing was perfectly clear, it was considered that the cream was almost completely free from milk-serum and the albuminoids it contained. In the washed cream there was no sign of butter being formed. Stirred in water, it formed an emulsion which, even under the microscope, could not be distinguished from fresh cream. If the fat globules in milk and cream consist of fat only, and are kept from coalescing by the peculiar consistency of the milk-serum, one would expect to see them adhere and form grains of butter when the milk-serum has been washed away. That the fat globules consist of something besides fat is also proved by the following experiment: The washed cream was shaken up with an equal volume of ether, when

only a small portion of the fat solution separated; the cream, however, changed into a gelatinous mass, which collected in the upper part of the vessel, and constituted about one-half of the volume of the mixture. On the addition of a few drops of acetic acid, and again shaking the solution, all the fat dissolved, and under the clear solution of the fat there remained a somewhat opalescent aqueous solution, containing a gelatinous precipitate in considerable amount. Just the same phenomena were observed on shaking the cream with benzene instead of with ether.

The attempts to wash the fat globules with water, which had proved unsuccessful, were repeated with more success by using a hand-separator ("Alpha-Baby"). Ten litres of fresh milk were separated by this machine, the cream mixed with 8 litres of water, and the mixture separated again; and in this way the fat globules were washed four or five times. It was found that at each separation the quantity of cream obtained, and its concentration, varied according to the temperature of the water used. The best result was obtained with water at 35° C. The cream obtained at each separation contained considerably less fat than the cream from the previous separation. In some of these experiments the cream was analysed after each washing in order to be able to calculate, from the composition of the cream and from the quantity of water which had been used, how much serum there would be left in the cream. It was found that after four washings the cream ought to be nearly devoid of milk-serum, for the calculation showed that there would be only 0·003 per cent. of albuminoids derived from the milk-serum present in it, or 0·02 parts to each 100 parts of fat. But the analysis of the cream revealed the presence of 0·184 per cent. of albuminoids, or 1·30 parts per 100 parts of fat; that is, sixty-five times as much as that derived from the milk-serum. Four washings were therefore considered sufficient to render the fat globules as free from milk-serum as could be effected by washing. I have made nine experiments with washing the cream either by water in a separator, or by means of sugar solution in a separatory funnel, and always found in the washed cream a large quantity of albuminoids, but most when the washing was done with sugar solution. As a considerable amount of the albuminoid is separated from the cream when this is washed in a cream-separator, I conclude that a certain proportion of the albuminoids is found in such a state of connection with the fat globules that it cannot be totally removed even by a thorough washing with water. In no case was there any formation of butter to be seen, and the fat could not be extracted by simply shaking with ether or benzene; this converted the cream into a gelatinous mass, in which the fat globules could be seen under the microscope apparently in an unaltered condition. By the addition of acetic acid and renewed shaking, the mixture would separate into a clear solution of fat and an opalescent liquid containing a slimy substance, which could be separated with difficulty by filtration. To obtain this substance, the following procedure is advisable: Shake the washed cream with an equal volume of strong alcohol, then add twice as much ether as the alcohol used, and a proportion of benzol. After thorough shaking, the clear fat solution will gather on the top, and underneath a tolerably clear alcoholic solution will be found, in which there is a gelatinous precipitate of albuminoid which can easily be separated by filtration. Washed with strong alcohol, and afterwards with ether, and dried in the air at the ordinary

temperature, it forms a loose, fine, hygroscopic powder of a grayish-white colour. The albuminoid prepared in this way has exactly the same properties as the albuminoid found in butter. It is insoluble in dilute ammonia, acetic acid, or hydrochloric acid; it swells considerably, without dissolving, in weak solutions of the alkalies. It is only partly soluble in dilute potassium or sodium hydrate. It gives the reactions peculiar to albuminoids, and, when heated with dilute hydrochloric acid, yields a substance which reduces Fehling's solution. The amount of copper reduced by 100 parts of the dry ash-free albuminoid is 6·5 parts. The dried and ash-free albuminoid contains 14·76 per cent. of nitrogen and 2·2 per cent. of sulphur. For comparison I give the proportions of nitrogen and sulphur found in the other fairly well-defined albuminoids:

	Nitrogen.	Sulphur.
Serum globulin, from blood of a horse	15·85	1·11
Serum albumin ,, ,, ,, ...	16·04	1·80
Fibrin	16·91	1·10
Albumin, from egg	15·25	1·80
Albumin, from cow's milk	15·77	1·73
Casein ,, ,, ...	15·70	0·80
Mucin, from salivary gland	12·32	0·84
New albuminoid from washed cream	14·76	2·20

It will be seen that this new albuminoid contains 1 or 2 per cent. less nitrogen than the albuminoids proper, and 2½ per cent. more nitrogen than mucin from the salivary gland; also that its content of sulphur is larger than that of the other albuminoids, from which it likewise differs in its insolubility in caustic alkalies and dilute acids. It is therefore claimed as a new and hitherto unknown albuminoid.

What is the connection between this albuminoid and the fat globules? I thought from the first that the albuminoid might form a membrane around the fat globules, and I succeeded in staining the washed fat globules with the aqueous solutions of different colours, and the fat globules remained stained even after the removal of the excess of staining liquid by thorough washing. It is necessary to employ a stain which does not coagulate the albuminoid; for instance, an ammoniacal aqueous solution of picrocarmine. Under the microscope it was found that the fat globules themselves were not coloured, but were surrounded by a stained envelope which had the same colour as the stain used. This goes to show that the albuminoid forms a membrane around the globules; it also explains (1) why the fat globules in the washed cream do not adhere together to form butter; (2) why ether does not extract the fat from the washed cream; and (3) why such cream is no easier to churn than ordinary cream; but that (4) when churned the butter is formed just in the same way as when normal cream is churned. These facts, therefore, prove that this albuminoid, which adheres so tenaciously to the fat globules that it cannot be separated by washing, forms a *mucoid membrane** around each globule.

To find the amount of *mucoid substance* surrounding the fat globules in cow's milk, I assume that all, or nearly all, of this substance is left when the cream is washed with a strong sugar solution, and that washing with water removes a con-

* By the term membrane is not meant a solid film or skin, but merely a layer, a semi-fluid, viscous envelope.

siderable part of it. In the washed cream I found 0·363 parts of nitrogen to each 100 parts of fat. The albuminoid in the membrane contains 14·76 per cent. of nitrogen; therefore 100 parts of fat will correspond to 2·459 parts of albuminoid.* The amount of water and ash in the mucoid membrane is calculated from the analysis of butter and butter-milk from the same churning, in the way explained in the beginning of this paper. From thirteen different churnings of absolutely fresh cream the percentage composition of the mucoid substance in the butter was found to be:

	Mucoid Substance in Butter from Fresh Cream.
Albuminoid	6·42
Ash	1·03
Water	92·55†
Total	100·00

It is probable that the mucoid membrane of the fat globules is not altered in composition by churning at the ordinary temperature. The composition given above can therefore be taken as the correct composition of the mucoid membrane at the ordinary temperature. Having found to each 100 parts of fat in the washed cream 2·459 parts of dry albuminoid free from ash, the amount of mucoid substance of the above composition corresponding to 100 parts of fat is calculated to be 38·4 parts. The proportion between fat and membrane in the milk-globules is therefore as 100 : 38, or the globules consist of 72·5 per cent. of fat and 27·5 per cent. of mucoid membrane. According to my determinations, the specific gravity of the milk-fat at 15° C. is 0·9335, and that of the mucoid substance of the above composition is 1·0228. The proportion between the volume of fat and the volume of mucoid membrane around the fat globules is, therefore $\frac{100}{0{\cdot}9335} \div \frac{38}{1{\cdot}0228} = 2{\cdot}883$. If the radius of the globules with the membrane is called ϕ, and the radius of the fat globules proper is called r, we have:

$$\frac{r^3}{\phi^3 - r^3} = 2{\cdot}883; \text{ or,}$$
$$\phi = 1{\cdot}104r.$$

The thickness of the membrane $(\phi - r)$ is equal to 0·104 r, or about one-tenth of the radius of the fat globule. The specific gravity of the globule with the mucoid membrane is 0·9565, or appreciably higher than the specific gravity of the butter-fat.

The results of these investigations as to the structure of the fat globules in cow's milk enable us to explain several facts which have hitherto been somewhat inexplicable. It has already been mentioned that it is impossible to extract the whole of the fat from the milk on shaking out with ether, for the simple reason that the mucoid membranes prevent the ether from coming in contact with the fat of the milk

* It will be remembered that by washing with water only 1·30 parts of albuminoids were left in an experiment referred to above.

† It must be distinctly understood that this composition refers only to the mucoid in butter churned from fresh cream at the ordinary temperature. At raised temperatures water is given off.

globules. It has further been mentioned that the globules in washed cream, on being shaken with ether, form a voluminous gelatinous mass, no solution of the fat taking place, for the fat globules remain apparently unaltered in the jelly. This gelatinous mass is formed by the swelling of the mucoid membranes, which are able to absorb ether in a large amount. The mucoid substance surrounding the fat globules absorbs readily ether or benzene, and forms with these a perfectly clear jelly, and it is impossible to perceive small quantities of this swollen mucoid substance in the fat solution. This is of some importance in connection with several methods for estimating the amount of fat in milk.

In the *volumetric* process of Soxhlet the fat is extracted by shaking the milk with a certain amount of potassium hydrate and ether, and estimating the specific gravity of the ethereal solution of fat by means of a special apparatus. It is evident that the value of this method depends upon whether the fat is completely extracted from the milk, and whether the ethereal solution contains nothing but fat. Experience has shown that in some cases it is impossible to get the fat extracted completely from the milk, as a jelly is formed which collects on the top of the mixture, while very little clear fat solution is formed. At other times the method of Soxhlet yields too high a result, and this is evidently due to the circumstance that the fat solution contains some of the swollen mucoid substance, which raises its specific gravity. The same observations apply to the method described by Röse, in which the milk is shaken with ether and petroleum ether after the addition of a certain amount of ammonia, the fat solution being evaporated in a small tared flask, and the dry fat weighed. As the mucoid substance swells considerably under the influence of ammonia, it is very likely that the fat solution obtained by Röse's method always contains some mucoid substance. In experiments in my own laboratory, estimations of fat in milk gave in nearly every case a higher result by Röse's method than by that of extraction after complete drying with kaolin powder. On an average of 500 estimations, 0·04 per cent. more fat was found by Röse's method. In estimating the fat in butter-milk by Röse's method, a still greater discrepancy was found. From the average of the analyses of 82 samples of butter-milk, 0·15 per cent. more fat was found by Röse's method, the limits being 0·11 and 0·18. In the fat extracted by Röse's method a nitrogenous substance was proved to be present. This difference in the results of analysing milk and butter-milk by Röse's method can be shown to be in relation to the amount of mucoid substance in the milk and butter-milk. During the process of churning about three-quarters of the membrane is removed from the globules and left in the butter-milk. While, therefore, fresh milk contains about 1·2 per cent. of mucoid substance (containing about 6 per cent. of albuminoid), butter-milk contains 5 per cent. of mucoid substance, or four times as much as fresh milk; and the excess of fat found by Röse's method was just four times as great when analysing butter-milk as when analysing fresh milk. To extract the milk-fat alone by means of ether or petroleum ether, it is necessary to completely dry the mucoid membrane, as is done when analysing milk by Adams' and several other methods. Even where the milk is treated with strong sulphuric acid before separating, as in the processes of Babcock, Lindstrom, and Gerber, the fat is not pure. The fat, when cooled down until it has solidified and examined under the microscope, is found to

contain minute drops of water, similar to those found in butter. These drops are formed by mucoid substance which has not been separated from the fat.

The membranes around the fat globules prevent them from adhering. To make them adhere—to become butter—the membranes must be for the greater part removed, as is done by churning; but even after the fat globules have been united to form butter, it is likely that each globule is still surrounded by an extremely thin membrane, because many of the globules can again be separated by simply squeezing the butter to a very thin layer. In the edge of such a layer the microscope reveals, especially with dark stage illumination, a number of fat globules separated from the margin of the butter layer, which swim about in an isolated condition in the water that is squeezed out of the butter. The greater part of the membranes is removed during the churning, and when they have been sufficiently reduced, the fat globules adhere together and form small butter-grains with the remaining part of the membranes. These latter are enclosed by the globules, and when examined under the microscope in a thin layer of butter have the appearance of very minute drops of water. In this way the numerous very minute drops of water found in butter are formed; they consist, in fact, not of water, but of the watery mucoid substance. As the churning proceeds, the small butter-grains join together to form larger grains, thereby enclosing particles of butter-milk, which can be seen in the butter as larger drops. The larger particles of butter-milk may be removed by working the butter, but the microscopically small drops, partly formed by the mucoid substance enclosed between the fat globules, cannot be removed by any amount of working the butter. These small drops form an essential constituent of butter, and give it that characteristic appearance, consistency, flavour, and taste, which distinguish it from pure butter-fat. About one-half of the amount of water present in butter is derived from the amount of mucoid substance it contains, which, on the average, is present to the extent of 7 per cent. by weight. The peculiar structure of butter can be seen when a lump of butter the size of a pin's head is gently pressed between the slide and cover-glass to a thin layer without squeezing any of the aqueous portion out of it. Examined under a lower magnification of about 100 diameters, by ordinary illumination with central light, and with a suitable diaphragm, large liquid drops will be seen, and, besides these, a countless number of small spherules, varying in size from mere points to large drops (Fig. A in the plate opposite page 207). In the usual explanation of this microscopical appearance, the numerous very small spheres are regarded as fat globules. But this is not correct; all the spherules seen in a layer of butter when examined in this way are, without exception, liquid drops; the fat globules are absolutely invisible with this kind of illumination. If the layer of butter is submitted to further pressure, whereby the larger water-drops are squeezed out, its thickness can be reduced to about the one-hundredth of a millimetre or less. When this is examined under a magnifying power of about 500 diameters, and with ordinary central illumination, a large number of spherules, varying greatly in size and having a dark edge, will be observed; these are all separated from one another, leaving some vacant places (see Figs. B and C). They all evidently consist of the same substance, and, as many of them are much too large to be fat globules, and as they are all separated, none of them can possibly be fat globules. If the illumination is changed to dark-

ground illumination, the similarity between these spherules and fat globules disappears, and they are found to be water-drops, with a bright reflecting surface (see Figs. D and E). Their aqueous nature is also proved by the fact that many of them contain micro-organisms moving briskly about, and this is only possible in an aqueous liquid. Further, the drops are colored by aqueous solutions of coloring matters, but they remain uncolored when a thin layer of butter is treated with osmic acid.

It is not impossible, however, to see the fat globules in a thin layer of butter. By ordinary central illumination nothing but the liquid drops can be seen (Fig. C), but by dark-ground illumination it is possible, in a very thin layer, to see the fat globules as faint circles, very different in appearance from the luminous drops (Fig. D). The thinner the layer, the more distinctly can the fat globules be seen, and near the edge of an extremely thin layer of butter, with dark-ground illumination, they sometimes present an appearance like pebbles on the seashore (Fig. F, where only a few blurred drops are seen, the many faint circles being all fat globules). A comparison between the two Figs. E and F in the plate is very instructive. They are both taken under the same magnifying power (550 diameter), with the same form of illumination, and represent thin layers of butter. In E nothing is seen but liquid drops, evidently of the same nature as the larger drops seen in Fig. D, while in F hardly anything but fat globules.

If butter is melted by being placed in boiling water, the butter-fat does not collect as a clear fatty layer, but a cream-like layer is formed, in which the microscope reveals the same structure as in the original butter. When butter is melted, a white precipitate forms, which consists of the mucoid membranes, the butter-milk, and salt. If the fat is decanted, the precipitate, after being washed with ether, is seen, under the microscope, to consist of a countless number of microscopic watery drops. Butter, therefore, is a conglomerate of fat globules interspersed with a countless number of very minute liquid drops, of which the smallest and most numerous are formed by the mucoid substance originally coating the fat globules.

A comparison of Figs. D and E, both showing the liquid drops in a thin layer of butter with the same power (550 diameter), will show how various the size and number of these drops may be in different kinds or qualities of butter. I have made a very close study of this subject, and have found that in the best clear waxy butter the number of drops in a cubic millimetre is between three and four millions. In a kind of butter, which it is not desirable to make, being generally bitter and of an opaque or "thick" appearance, the number is on the average thirteen millions. This kind of butter is sometimes found as light streaks or patches in a clear waxy yellow butter. Fig. D shows the structure of the clear waxy butter, Fig. E of the "thick" butter. Some butter is apt to give off brine. This is not necessarily a proof that it contains a large amount of water, but I have found that in such butter some very large drops are generally present (Fig. D). These are removed when the butter is carefully worked, but no amount of working will remove the very small drops. The "thick" butter may sometimes contain as much as 18 per cent. of water in small drops. This water cannot be removed, and the butter appears dry, while a "clear" butter, containing less than 14 per cent. of water, some of which is present as large drops (as in Fig. D), may appear wet and give off brine.

I have found that this different structure of butter—the difference in the size and number of these liquid drops—is caused by changes in the mucoid substance, brought about by the action of special micro-organisms during the ripening of the cream. Some bacteria which cause milk to turn bitter often make the butter "thick," *i.e.*, give it a large content of small liquid drops.

RESUMÉ.

The fat globules in milk are coated with a mucoid substance forming a membrane around each globule. These membranes or semifluid envelopes are retained when the fat globules are washed free from milk-serum. The membranes can be stained and seen under the microscope. The mucoid substance of the membranes is found also in butter as minute liquid drops, and forms over 60 per cent. of the total albuminoid content of butter. It can be proved by a simple calculation from the composition of butter-serum and butter-milk-serum that butter-serum contains an aqueous albuminoid. The albuminoid in question differs from the albuminoids hitherto known.

Butter is a conglomerate of the fat globules of milk, interspersed with numerous minute liquid drops, of which the smallest consist of mucoid substance, the largest of butter-milk. The structure of the butter and the number of liquid drops it contains is partly dependent on the kind of micro-organisms present during the ripening of the cream.

PHOTOMICROGRAPHS OF THIN LAYERS OF BUTTER. BY PROF. V. STORCH.

Power: Fig. A × 93 diameter; Figs. B to F × 550.
Figs. A, B, and C, by central illumination.
Figs. D, E, and F, by dark-ground illumination.

FIG. A.—Some large drops of butter-milk are seen. All round bodies down to the smallest specks are drops of butter-milk and mucoid substance. Thickness, 0·022 mm.

FIG. B.—Liquid drops in butter, the larger drops have been squeezed out. Thickness, about 0·01 mm.

FIG. C.—Liquid drops in a "clear" butter, which gives off brine. Thickness, 0·01 mm.

FIG. D.—The same object as in C, with dark-ground illumination. Some fat globules are seen in the dark spaces between the luminous water-drops.

FIG. E.—Liquid drops in "thick" butter. Thickness, 0·008 mm.

FIG. F.—Fat globules in the thin edge of a very thin layer of butter.

DISCUSSION.

The PRESIDENT having invited discussion,

Mr. RICHMOND said that, owing to the courtesy of Mr. Faber, he had had the advantage of reading through Professor Storch's paper. He had also been performing some experiments, though he was grieved to say that, on account of ill-health, he had not been able to make these experiments as extensive as he could have wished

them to be. On certain points he was able to confirm what Professor Storch had said. He had succeeded in separating the substance described by a slight modification of the method given in the paper. He had taken cream, and washed it with cane-sugar solution until all the milk-serum was washed out. Treatment with alcohol, ether, and benzene produced a substance agreeing in its properties with the substance described. He had also taken butter-milk, and passed it through a separator, obtaining a large deposit. He had not been able to estimate the quantity of this deposit, nor to make an extended examination of it, but it seemed to correspond in its properties with the substance described. Microscopical examination showed that the substance obtained from butter-milk was not in a state of absolute purity. It contained small quantities of vegetable matter (probably derived from the dust of the air) and cells of the udder, which were always present in the deposit found on the inside of the drum of a separator. It also contained a substance having somewhat the appearance of membranes which had been burst, and consisting of partially globoid forms, which appeared to have undergone considerable rough treatment. Innumerable micro-organisms were present in these globules. It could hardly be expected that this substance would agree in all its properties with that described by Professor Storch, owing to the presence of the micro-organisms, which would probably cause a certain amount of change. There were certain points on which he could hardly agree with Professor Storch. If around the fat globule there was a membrane the proportion of which was 38·5 to 100 of fat, it might be expected that cream containing 50 or 60 per cent. of fat would contain a large quantity of this membrane. For instance, cream containing 50 per cent. of fat might be expected to contain about 19 per cent. of the membrane, leaving 31 per cent. of milk-serum. He had therefore thought that by estimating the milk-sugar and proteids in cream, one should get conclusive evidence as to its presence or absence. He had accordingly made a few analyses of cream, a typical one of which was as follows:

Water	39·37
Fat	56·09
Milk-sugar	2·28
Proteids	1·57

Assuming that all except the fat had the same composition as the separated milk obtained at the same time (neglecting the small quantity of fat contained by the latter), he had calculated that it should contain 2·23 per cent. of milk-sugar and 1·55 per cent. of proteids; whereas if Professor Storch's theory were correct, the figures should be about half those amounts. He had found in the case of other samples of cream that the estimated milk-sugar agreed with that calculated from the composition either of the milk or of the separated milk obtained at the same time. The analysis given above was made on the actual sample from which the substance was prepared, so that it must have been present in the sample; but no evidence was given by these figures that it was present in the quantity indicated by Professor Storch, or that the membrane was composed of an albuminoid having the composition stated in the paper. He thought that the evidence advanced by Professor Storch as to the presence of a membrane was not altogether conclusive. The two points relied on by Professor Storch were, firstly, the staining round the fat globules;

and, secondly, their behaviour with ether. It was quite possible to explain these points without assuming the presence of a membrane. If the physical properties of very small globules were considered, it would be seen that the pressure on the surfaces of these globules must be large, and that there must be practically a condensed layer of liquid around them. It seemed to him quite reasonable to suppose that if a stain got into this layer it would not be easily washed out, even though the layer might not necessarily be solid or semi-solid. It also seemed to him that a mixture of small globules of fat with water must be considered as a mixture of two liquids which were not soluble one in the other, separated by a semi-permeable membrane. It should be pointed out that no material membrane need exist; the surface between water and air was almost a perfect semi-permeable membrane, yet no actual membrane was present. The quantity of ether dissolved in the fat was proportional to the quantity dissolved in the water, and to the difference between the maximum solubility of ether in water and in fat. The fat in milk was almost wholly in a solid condition, a condition by no means favourable for dissolving ether; and therefore it seemed quite possible to explain the phenomena observed by Professor Storch when cream was treated with ether, without assuming the presence of a membrane. With regard to Professor Storch's further arguments based on the evidence furnished by the difference between Storch's own method and that of Röse, it need only be pointed out that butter-milk differed in many respects from milk to show how purely circumstantial this evidence was. The appearance of water-globules in the fat separated in the Babcock and other processes when cooled was no argument as to the presence of a membrane; the known solubility of water in hot fat had apparently been overlooked. In conclusion, his opinion was that Professor Storch had established the presence of a new albuminoid in milk, but that the view that he adopted as to its forming a membrane round the globules appeared to be insufficiently supported by the evidence obtained and in contradiction to some of the known facts. He might throw out a suggestion that Professor Storch's albuminoid might be the protoplasm of the cells in which the milk was secreted.

Mr. HEHNER said that the question which this paper revived was one which had seemed to be practically decided in favour of the non-existence of a coating on the fat globules. No doubt very many phenomena connected with the physical properties of milk were more readily explicable if the fat globules were assumed to be coated, but he must say for himself that he was not at all convinced that the fat globules were really coated. The substance described was a very extraordinary substance, differing from any albuminoid known. He would have liked to see actual microscopical representations of the isolated substance; being, as he understood, quite insoluble in most reagents, it ought to retain its characteristics more or less when isolated. He would like to know whether the substance was acted upon by gastric juice, and could be dissolved in this way from the fat globules. Being an albuminoid, it would probably be peptonized, and in that case the fat globules would run together at once without any mechanical treatment if it were really only the presence of an albuminoid substance which prevented their cohesion, instead of the physical causes to which it was attributed by those who contended that the fat globules became connected. He would also suggest that the production of a considerable quantity of Fehling-reducing

substance by hydrochloric acid might be due to the cellulose which would undoubtedly be present, seeing that there would be numerous micro-organisms present containing cellulose, which when acted upon by hydrochloric acid should yield sugar.

Mr. A. C. CHAPMAN remarked that it was well known that certain albuminoids possessed the property of yielding, on boiling with hydrochloric acid, a substance reducing Fehling's solution. He had himself found that such albuminoids existed in yeast.

Mr. ALLEN said it was a well-known fact that bodies of the mucin class had a constitution allied to that of the glucosides, being compounds of a proteid with animal gum, which latter body yields a reducing unfermentable sugar by boiling with dilute sulphuric acid.

Mr. FABER said it was gratifying to find that Mr. Richmond had succeeded in preparing the substance in question. The most important point seemed to be the actual existence of the body. Mr. Richmond had found that it did exist, but had failed to find it in so large a quantity as Professor Storch. In trying to find it, however, he had used a method slightly different from that used by Professor Storch. Professor Storch had tried to produce the albuminoid from very thick cream, but had found that this method did not answer. He had concluded, differently from Mr. Richmond, that his failure to find it in the thick cream was due to the fact that it had been separated from the fat. Not being a perfectly solid body, it was not at all unlikely that during the process of separation it was removed from the fat globules. Its specific gravity was appreciably higher than 1, and that of the butter-fat being less than 1, it was not at all unlikely that during the treatment in the separator these two semi-fluid bodies should separate. It was to be pointed out that the membrane was not, as Mr. Richmond seemed to consider, a solid membrane. It was a semi-fluid, and the difference between a condensed layer of something else and the semi-fluid mucoid substance was therefore very small, and the possibility of explaining the phenomenon in a different way was, he thought, hardly sufficient to completely discount Professor Storch's explanation. Mr. Hehner was not convinced of the existence of this peculiar substance, or, at any rate, of its presence as a membrane, and asked if it was not possible to ocularly prove its existence. When the cream was churned and the mucoid substance broken away from the fat globules, some of it remained enclosed with the fat globules, forming small mucoid drops. These drops retained their shape. If butter were melted at a low temperature, a precipitate was formed under the clear butter-fat, and if this precipitate were rinsed with ether or benzene and examined under the microscope it would be found to consist of these watery or mucoid drops, which retained their shape, and did not run together, as would have been the case in an aqueous solution of an albuminoid. The question as to whether a reducing substance could be formed from the cellulose surrounding the micro-organisms in the cream was one to which, he regretted to say, he felt unable to reply. Assuming, however, that the micro-organisms were actually surrounded by cellulose, it would seem that if their number was such that the cellulose coating them could reduce 6½ parts of copper per 100 of fat, it would be the easiest thing in the world to see them, and he could hardly imagine that Professor Storch would have failed to notice them. That reducing substances were formed from other

albuminoids on boiling with hydrochloric acid was of course well known to Professor Storch, and was duly mentioned in the paper. Of the albuminoids proper, serum globulin gave off a reducing substance, though in smaller quantity than the body under consideration; and similar reducing substances were formed in considerable quantities from compounds of the mucin group.

ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

FOOD AND DRUGS ANALYSIS.

Reagents distinguishing between Raw and Boiled Milk. R. Dupouy. (*Bull. de la Soc. Pharm. Bordeaux*, March, 1897; through *Jour. Pharm. Chim.*, 1897, 397, 398.)—*Guaiacol.*—One c.c. of a 1 per cent. aqueous solution is added to an equal volume of the milk, and then one drop of ordinary commercial hydrogen dioxide. An immediate yellow colour is produced in the case of raw milk, while with boiled milk the mixture remains colorless.

Hydroquinone.—One c.c. of a freshly-prepared 10 per cent. aqueous solution, mixed with 3 c.c. of raw milk and fifteen drops of commercial hydrogen dioxide, gives an immediate rose coloration, and the mixture, after standing for three or four minutes, deposits green crystals of quinhydrone, $C_6H_4O_2 \cdot C_6H_4(OH)_2$.

Pyrocatechin.—One c.c. of an aqueous 10 per cent. solution, mixed with an equal volume of raw milk, produces, after the addition of a drop of hydrogen dioxide, a yellowish-brown colour.

α-Naphthol.—An aqueous solution, mixed with raw milk and a drop of hydrogen dioxide, gives a violet-blue colour. β-Naphthol treated in the same way produces no colour. Hence this reaction distinguishes between the two naphthols.

Paraphenylene diamine.—Several centigrammes are dissolved in 1 c.c. of water, and to the cooled solution an equal volume of milk and one drop of hydrogen dioxide are added. If the milk is raw, a very dark violet colour is produced. This reaction is very sensitive, and can be used with watered and skimmed milks, which, with the preceding reagents, give the colorations, but with less intensity than normal milk (see also ANALYST, this vol., p. 104). C. A. M.

On the Albuminoids of Cow's Milk. K. Storch. (*Monatshef. Chem.*, 1897, xviii., 244-281.)—The author claims that his experiments confirm Hammarsten's conclusion that there is only one kind of casein present in cow's milk. On saturating milk with sodium sulphate, magnesium sulphate, or sodium chloride, this casein is not precipitated unchanged, but is decomposed into two bodies of similar composition, one of which is precipitated. The casein can be precipitated in an unaltered form by means of acetic acid, and it is possible that simultaneous saturation with two of the three above-named salts might also cause it to separate without decomposition.

C. A. M.

On the Cause of the Coagulation of Heated Milk. B. Bardach. (*Monatshef. Chem.*, 1897, xviii., 199-216.)—The author's investigations have led him to the conclusion that the view of Cazeneuve and Haddon that the coagulation of heated milk is due solely to the action of the acids produced by the oxidation of lactose is incorrect. Preliminary experiments showed that the times and temperatures required for the complete coagulation of fresh milk were: At 100°, about twelve hours; 110°, five hours; 120°, one and a half hours; 130°, one hour; 140°, twenty minutes; 150°, three minutes. The partial separation of albuminoids which occurred on heating milk for several hours at a little below 100° was regarded by the author as quite distinct from coagulation proper. Throughout the subsequent experiments the time and temperature adopted were one hour and 130° respectively, and the milk was heated in an autoclave to avoid frothing.

Fresh milk coagulated at this temperature, and the clear serum, when distilled, yielded as the result of many determinations not more than 0·003 gramme of formic acid per 100 c.c. On adding 3 c.c. of 20 per cent. sulphuric acid to the coagulated milk, the amount of formic acid increased to 0·03 gramme. A subsequent determination of the volatile acid by distilling fresh milk with the same proportion of sulphuric acid gave 0·004 gramme. Hence the difference between this result and the preceding one is ascribed to the volatile acids liberated from combination by heating milk at 130°. This amount of acid is too small to produce coagulation in the cold, for which an acidity of 0·13 per cent. (calculated as acetic acid) was required (Hammarsten, 0·075 to 0·1 per cent.). As regards the combined action of heat and acid, fresh milk, boiled with increasing small amounts of acetic acid, first showed signs of coagulation with 0·055 per cent.; but coagulation was not complete until the percentage reached 0·07—an amount considerably in excess of that produced by heating fresh milk at 130°. Therefore the possibility of the volatile acid being the sole cause of coagulation was out of the question.

A 5 per cent. aqueous solution of lactose heated for an hour at 130° still showed a neutral reaction; but on heating the solution with 0·5 per cent. of hydrogen disodium phosphate at 100° or 150° the liquid became acid. Equal volumes of fresh milk and an aqueous solution of lactose containing hydrogen disodium phosphate, and previously heated at 130°, were then mixed, but there was no alteration in the milk, even when heated on the water-bath for a short time, or boiled once. Hence neither the volatile nor the total acids produced by the oxidation of lactose can be taken as the exclusive cause of coagulation.

The behaviour of solutions of pure casein was next studied. Eight grammes were dissolved in 80 c.c. of water containing 0·8 gramme of hydrogen disodium phosphate, and the solution heated for an hour at 130°. A partial coagulation occurred, but a further deposit could be obtained from the filtrate on the addition of acetic acid. In a solution of half the strength coagulation was retarded, and in very dilute solution was altogether absent. A solution of casein in dilute sodium hydrate subsequently neutralized with hydrochloric acid behaved in a similar manner, showing only incipient coagulation. But a solution of 4 grammes of casein in 110 c.c. of water containing 0·4 gramme of hydrogen disodium phosphate, mixed with an equal volume of 10 per cent. lactose solution, completely coagulated.

A similar solution of casein heated for an hour at 130° gave, as before, incipient coagulation. The coagulum was filtered off, and the filtrate mixed with an equal volume of lactose solution containing hydrogen disodium phosphate, and previously heated at 130°. On now boiling the mixed solution, the remaining casein was precipitated, and the coagulum and filtrate appeared in every respect like those obtained with milk. From these facts, the author concludes that the coagulation of milk on heating is due to the alteration of the casein by heat into such a state that it can be precipitated by the small amount of acid derived from the lactose, which is otherwise incapable of affecting it. C. A. M.

Estimation of Fat in Separated Cream. M. Weibull. (*Chem. Zeit.*, 1897, xxi., 333.)—This process is based on the fact which had been previously indicated by Richmond, that the ratio of water to solids-not-fat is the same in separated cream as in milk. This ratio for the cows usually bred in Mid and North Germany, and in South Sweden he takes at 8·7 ; and his formula for calculating the fat is $F = 1{\cdot}1\,T - 9{\cdot}5$. A number of analyses of sweet and sour cream are appended, showing a mean difference of 0·5 per cent. (maximum 0·9) between the amounts of fat obtained by extraction and by calculation ; and as he lays down the principle that for the commercial examination of cream a process is sufficiently exact if it gives results within 1 per cent. (or at the most 0·5 per cent.) of the truth, he holds that the method is perfectly suited for ordinary purposes. In the case of cream adulterated with water, the process causes the sample to be returned somewhat below its actual value—a point rather in its favour. Creams obtained by the old method of skimming do not yield quite such concordant results ; but of twenty-five specimens only two gave differences of 2 per cent. A lengthy table calculated from the above formula is also included in the paper. F. H. L.

Identification of Genuine Butter, and a Method for distinguishing between Different Fats. A. von Asboth. (*Chem. Zeit.*, 1897, xxi., 312.)—For the examination of butter the author determines in the usual manner the foreign ingredients—water, casein, and salt—the melting-point, and the free acids ; but he obtains the Köttstorfer, Reichert, and Hehner numbers in a different way. His process depends on the fact that the lead salts of the soluble fatty acids are also soluble in water, while those of the insoluble acids are equally insoluble. 2·5 grammes of the sample are heated with 25 c.c. of semi-normal alcoholic potash, and the excess of alkali is titrated with *acetic acid* of equivalent strength to determine the saponification number. The residual liquid is then washed into a 300 c.c. stoppered cylinder containing 150 c.c. of water and 30 c.c. of a 10 per cent. solution of lead acetate, and the whole is well shaken until the precipitate adheres together and the liquid has become clear. It is next thrown on a filter of thick strong linen, and both the cylinder and precipitate washed with cold water. The lead salts of the insoluble acids are freed from moisture by squeezing them thoroughly, removed from the cloth, and put back into the cylinder. 200 c.c. of ether are added, and the mixture agitated till the solid mass is completely broken up. It is rinsed into a 250 c.c. oil burette,

excess of 1 : 4 HCl introduced, and shaken until the ethereal layer becomes clear. The acid is then run off, and the ether washed with water till neutral. The volume of the liquid is read, 50 c.c. are evaporated and dried to constant weight, and another 50 c.c. evaporated, dissolved in 50 c.c. of alcohol, and titrated with potash. The Hehner number is derived from the weight of the dry residue; while the figure obtained on titration, deducted from the saponification number, gives the Reichert number. The lead precipitate does not adhere to the filter-cloth, but may be easily and completely removed by the fingers or a knife; and the whole process is exact, free from manipulative difficulties, and can be carried out—except the drying of the acids—in an hour.

The author's method for distinguishing between different fats is based on an attempt to identify each variety by the proportion of oleic acid it contains. Three grammes of the sample are saponified in 50 c.c. of alcohol and 1 or 2 grammes of caustic potash. Phenolphthalein is added, and the solution neutralized with acetic acid. It is then treated with lead acetate as above, and the insoluble lead soaps collected on the linen filter. The cylinder is drained from the water, the precipitate and 150 c.c. of ether put in, shaken up, and allowed to rest over-night. A funnel and a thick filter-paper large enough to hold the solution and the washings are placed in the oil burette, moistened with ether, the contents of the cylinder thrown thereon, and covered with a dial-glass. The insoluble matter is washed three times with ether—using not more than 100 c.c. altogether—in order to make certain that the whole of the lead oleate is dissolved. The filtrate in the burette is acidified with HCl, shaken up, and washed as before. The total volume of the ethereal liquid is read off, 50 c.c. are evaporated in a beaker on the water-bath, taken up in alcohol, and titrated with decinormal acid. The amount of alkali used multiplied by 0·0282 gives the oleic acid in the ether, whence may be found the quantity existing in the fat. Examined in this manner, ten different samples of butter gave 33·72 to 37·40 (mean 34); several margarine butters, 45·48 to 46·0; oleomargarines, 42·61; baker's fats, 52·73 to 53·37; six lards, 56·91 to 58·08; six goose fats, 64·97 to 67·29; one mutton tallow, 25·44; four beef tallows, 33·03 to 33·95; and two "market fats," 47·76 to 48·69 per cent. of oleic acid respectively. It will be seen that the figures for each separate substance agree closely, except in the case of butter itself (and here the variation is not serious); and the values of all the other fats are very different, with the sole exception of beef tallow, which, however, may be readily distinguished from genuine butter by its difference in consistency and melting-point.

The author holds that butter may be identified by determination of the melting-point, saponification, Hehner, and Reichert numbers; but when it is a question of what or how much foreign fat has been added, the new process, in conjunction with a qualitative examination, is distinctly preferable to any other.

F. H. L.

Properties of Oil of Egg. **M. Kitt.** (*Chem. Zeit.*, 1897, xxi., 303.)—The oil was prepared by extracting with ether the hard-boiled and dried yolks of the (hens') eggs, evaporating the solvent, and removing the lecithine and other suspended matter by filtration through a hot funnel. Forty eggs gave 682 grammes of dry yolk, con-

taining 131 grammes of pure oil, or 19 per cent. It has an orange-yellow colour, is partially solid at ordinary temperatures, gives the Hager-Salkowski's reaction for cholesterin, and yields a solid elaidin. Its constants are as follows:

Oil.		Fatty Acids.	
Specific gravity at 15° ...	0·9144	Melting-point	36-39° C.
Acid number...	1·2	Saponification number (mean)	194·9
Saponification number (mean)	190·2	Iodine number (mean) ...	73·7
Ether number	189·0	Acetylated acid number ...	189·7
Iodine number	72·1	Acetylated sapon. number...	201·6
Hehner's number	95·16	Acetyl number	11·9
Reichert-Meissl number ...	0·4	Mean molecular weight ...	285·0
Glycerin, per cent.	10·4		
Lecithine, per cent.	0·2		
Cholesterin, per cent. ...	1·5		

The lecithine was determined by fusing the fat with potash and nitre, estimating the phosphoric acid in the residue with molybdate, and multiplying the amount found by 11·366. Using Benedikt's formulæ for the calculation of the fatty acids, these become:

Oleic acid	81·8 per cent.
Palmitic acid	9·6 ,,
Oxy- (? stearic) acid	6·4 ,,
Stearic acid	0·6 ,,
Cholesterin	1·6 ,,
	100·0

The oleic acid in the oil itself is therefore 81·3 per cent., or, calculated from the iodine absorption of the fat, 83·6 per cent.

F. H. L.

The Detection of Arachis in Chocolate. A. Bilteryst. (*Bull. de l'Ass. belge*, 1897, x., 447-452.)—According to the author, roasted earthnuts are frequently mixed with cocoa in the manufacture of chocolate, especially in Spain. In such cases a microscopical examination of the chocolate freed from fat and sugar will detect arachis starch, which has characteristic voluminous granules which can readily be measured. A portion of the sample (free from fat and sugar) should also be examined after treatment for twenty-four hours with a mixture of 50 grammes of chloral hydrate and 40 grammes of water. This often enables one to detect the characteristic cellular structure of the earthnut.

The results of the microscopical examination may be corroborated by an examination of the fat and determination of the albuminoid matters. The fat is extracted with carbon tetrachloride, and the refractometer reading taken. When the chocolate has been mixed with earthnuts useful indications may thus be obtained, but when earthnut-cake has been employed it is not possible to draw any conclusion.

The following results obtained by the author show to what extent a determination of the albuminoid substances by Kjeldahl's method indicates an admixture:

	Albuminoid substances Per Cent.
Pure chocolate	9·10
Earthnuts	28·18
Pure cocoa	17·57
Earthnut-cake	46·90
Pure chocolate + $\frac{1}{10}$ earthnuts	12·53
Pure cocoa + $\frac{1}{10}$ earthnut-cake	21·18
Pure chocolate + $\frac{1}{10}$ earthnut-cake	15·70

C. A. M.

The Composition of Chestnuts. M. Balland. (*Jour. Pharm. Chim.*, 1897, v., 525-527.)—In France chestnuts form a considerable item in the food of the people. In 1894, 2,963,845 cwt. were gathered, most of which must have been consumed in the country, since the exportations only slightly exceed the importations. In 1895 the amount imported (chiefly from Italy) was 5,963,273 kilos, as against 8,860,772 kilos exported. Of this latter quantity 5,922,018 kilos were sent to England, and nearly a million kilos to Algiers and Tunis.

The mean weight of the French chestnut was found by the author to be 18·60 grammes, while those from Naples and the Pyrenees were larger. The weight of the shelled nut was from 72 to 84 per cent. of the total weight. The following was the percentage composition of the shelled nuts:

		Water.	Nitrogenous substances.	Fat.	Sugars and Starches.	Cellulose.	Ash.	Acidity.
Minimum	normal	52·80	2·01	·0·45	31·54	0·74	0·57	0·059
	dried	0·00	4·45	1·17	82·17	1·76	1·24	0·164
Maximum	normal	62·60	4·31	1·73	40·74	1·36	1·22	
	dried	0·00	11·05	3·74	88·61	3·29	3·06	

The ash was not fusible like that of wheat; it contained less phosphates, but more chlorides and sulphates. It was usually more or less green, owing to the presence of manganese.

Roasted chestnuts as sold in the streets of Paris still contained 40 per cent. of water, while those cooked for the table by boiling in water contained 72 per cent.

C. A. M.

Detection of Artificial Coloring-matter in Sausages. H. Bremer. (*Forsch. Ber.*, 1897, iv., 45-48.)—The author has frequently met with cases in which the artificial coloring-matter could be detected microscopically in the fibre, but could not be extracted by any of the usual solvents, such as alcohol, amyl alcohol, or a mixture of glycerin and alcohol. In such cases a mixture of equal parts of glycerin and water, as recommended by Klinger and Bujard, is the best solvent. The finely-divided sausage is heated for several hours on the water-bath with two volumes of this mixture (slightly acidified), the yellow solution freed from fat and filtered, and the coloring-matter precipitated as lake by the addition of alum and ammonia. On placing the test-tube before the spectroscope, the absorption-lines of carmine lake lying between *b* and D may then be identified. Since the acid solution of the sausage coloring-matter is yellow, whilst carmine lake gives a red solution with hydrochloric,

nitric, and tartaric acids, it is suggested that the carmine is present in some other form than lake, possibly combining with the preservative to form a compound insoluble in alcohol.

In one instance in the author's experience a cervelat sausage thus colored had all the appearance of good meat when cut, but on further examination was found to be quite unfit for food, the acidity number of its fat being 76·0. C. A. M.

Estimation of Potassium Bitartrate in Wine. L. Magnier de la Source. (*Ann. de Chimie Analyt.*, ii., 101-103.)—To increase the accuracy of the evaporation method, which, though admittedly defective, is still generally employed, the author proposes the following modifications :

1. Ten c.c. of the wine are left to evaporate spontaneously in a basin placed in a desiccator over sulphuric acid, the operation requiring about three days for completion. Meanwhile a solution of potassium bitartrate (1 gramme per litre, the water containing 10 to 12 per cent. of alcohol) is prepared and supersaturated with potassium sulphate. By the aid of 10 c.c. of this solution, the dry residue of the wine is all dissolved, except the potassium bitartrate and sulphate contained therein, and after washing these crystals with the same solution (preventing as far as possible their escape from the basin on to the filter) they are dissolved in hot water, the filter being also washed with boiling water to dissolve any bitartrate thereon, and the resulting solution titrated with lime or baryta water, standardized to saturate not more than 0·005 gramme of bitartrate per 1 c.c. in presence of phenolphthalein.

The advantage presented by the solvent employed over the ordinary saturated solution of bitartrate is that with a small proportion of alcohol it contains only 0·3 gramme of bitartrate per litre, thus minimizing the effect of the errors due to evaporation on the filter, and the acidity of the liquid impregnating the latter.

2. One and a half grammes of potassium sulphate are added to 100 c.c. of wine, the liquid being rapidly concentrated to 15 c.c. on the water-bath, and left to crystallize for forty-eight hours in a cool place. The mother liquor is then decanted, and the crystals and filter washed with the same solution as in the first method, the titration being performed in a similar manner but with a stronger baryta-water (1 c.c. = 0·025 gramme of bitartrate), which increases the precision of the end-point.

The mother liquor, being saturated with potassium sulphate, cannot dissolve more than 0·4 gramme of bitartrate per litre (*i.e.*, 0·06 gramme per 15 c.c.), so that the error due to this cause is minimized.

The method is inapplicable to wines containing any notable quantity of ethyl-tartaric acid, or those wherein the initial proportion of potash does not exceed the amount required to convert the tartaric acid present into bitartrate. The latter case is of rare occurrence in ordinary wines. C. S.

Rapid Method of Detecting Lead Chromate in Papers used for Wrapping Eatables. J. Wolff. (*Ann. de Chimie Analyt.*, ii., p. 105.)—Chocolate, bonbons, etc., are frequently wrapped in paper coloured with lead chromate. To detect this

substance a strip of the paper about 5 c.c. square is moistened with 90° alcohol, and, the excess being poured off, a few drops of nitric acid are spread on the surface of the paper, which turns green from the production of chromium sesquioxide, aldehyde being evolved. The treated paper is washed with 10 to 15 c.c. of water, and this tested with a 10 per cent. solution of potassium iodide. If lead be present, a precipitate of iodide will be formed—provided the excess of nitric acid is not too great—whereas zinc gives no precipitate.

The method is also applicable for papers colored green by lead chromate and Prussian blue. The evolution of aldehyde, detected by its odour, proves the presence of chromium; lead is tested for as above, and the Prussian blue by the ordinary reagents. C. S.

The Presence of Starch and Strontium Sulphate in Opium. L. F. Kebler and C. H. La Wall. (*Amer. Jour. Pharm.*, 1897, lxix., 244-250.)—During the past two years the author has frequently found wheat starch in specimens of opium, varying in amount from a trace to 8 per cent. The adulteration is most frequently met with in Persian opium, and as this is exported by way of Trebizonde to Constantinople, and is there worked up to imitate Asia Minor opium, the admixture is probably made during the process.

The starch may be determined microscopically or chemically. In the former case 1 gramme of the opium is dried, triturated in a mortar with 2 c.c. of alcohol, and mixed with 8 c.c. of simple syrup. The starch granules are then counted in several drops of this with the aid of an ocular micrometer, and the average noted. A syrupy mixture of the same starch as that contained in the opium (say 1 per cent.) is prepared, and the number of granules in a drop of this determined in the same way. When by dilution or by making a more concentrated starch preparation a mixture is obtained containing exactly the same number of granules as the syrup from the sample of opium, the percentage of starch in the latter is readily calculated.

In the chemical method 10 grammes of opium are exhausted with cold water, and the residue boiled for fifteen minutes on the water-bath with 200 c.c. of alcohol containing 5 per cent. of potash. The liquid is filtered hot, and the residue washed with hot alcohol, and, after evaporation of all traces of the latter, boiled for three hours under a reflux condenser with 200 c.c. of water and 16 c.c. of hydrochloric acid (specific gravity 1·16). The contents of the flask are cooled, neutralized with sodium carbonate, filtered, and made up to definite volume. The amount of reducing sugars in an aliquot portion of the filtrate is determined gravimetrically or volumetrically, and the result multiplied by 0·9 gives the quantity of starch (including pentosans and other carbohydrates capable of hydrolysis by boiling with hydrochloric acid) in 10 grammes of opium.

Another impurity in opium is strontium sulphate; it can be estimated in the ash. The author considers its presence particularly objectionable, since it causes the percentage of morphine to appear considerably higher than is really the case. It was first noticed about a year ago, and the practice is still continued, some of the samples assayed during the past few months having yielded unusually high results when examined by the United States Pharmacopœia process. C. A. M.

Examination of Codeine Salts. K. Tambach and F. Henke. (*Pharm. Central. H.N.F.*, 1897, xxxviii., 159; through *Chem. Zeit. Rep.*, 1897, 107.)—Codeine itself contains 1 molecule of water of crystallization, and dissolves in 118·35 parts of water at 15° C.; the hydrochloride contains 1 H_2O, and dissolves in 25·88; the phosphate contains $1\frac{1}{2}$ H_2O, and dissolves in 3·2; and the sulphate 5 H_2O, and dissolves in 33·3 parts of water. The officinal test that the base or the phosphate should dissolve in strong sulphuric acid without colour must be carried out as follows: 0·1 gramme of the phosphate or 0·07 of the alkaloid is gradually added to 10 c.c. of sulphuric acid absolutely free from nitric or nitrous acids, and well stirred up. The pink colour which is first produced disappears in one or two minutes, and the liquid must then remain colourless. The presence of narcotine gives a greenish-yellow, afterwards an orange solution; with 0·1 per cent. the colour is reddish-violet. More than 2 per cent. of papaverine also yields a red-violet liquid. Morphine is indicated if a blue colour is produced when 10 c.c. of a solution of potassium ferricyanide, containing one drop of ferric chloride, are mixed with 1 c.c. of normal nitric acid in which 0·01 gramme of codeine has been dissolved. F. H. L.

The Chemical Valuation of Antipyrin. C. Kippenberger. (*Zeit. anal. Chem.*, 1896, xxxv., 659-677.)—When an aqueous solution of antipyrin, whether containing acid or not, is mixed with a solution of an iodine salt containing free iodine, a compound having the composition $C_{11}H_{12}N_2O.HI.I_2$ is precipitated. The reaction is quantitative when there is a sufficient excess of iodine, and when some mineral acid is present, can be used for the separation of antipyrin from phenacetin, sulphonal, aniline salts, and acetanilide.

A solution of antipyrin (as concentrated as possible) is shaken with a slight excess of iodine solution, prepared by mixing the ordinary $\frac{N}{10}$ or $\frac{N}{20}$ iodine solution with hydriodic acid in such proportion that 100 c.c. of $\frac{N}{20}$ iodine solution contain 4 c.c. of hydriodic acid of specific gravity 1·7 (= 52 per cent. HI). After from two to four minutes the liquid is filtered through asbestos from the precipitate, and an aliquot portion of the filtrate titrated with standard thiosulphate. From the amount of iodine consumed, the quantity of antipyrin can be calculated, 21·3 c.c of $\frac{N}{10}$ iodine solution corresponding to 0·1 gramme of antipyrin. The presence of hydriodic acid and its salts causes the antipyrin periodide compound to slightly dissolve, so that when absolutely accurate results are required the iodine solution should be standardized on an antipyrin solution of known strength.

Aniline salts also combine with iodine, but the compound is dissolved by acid. When they are present, however, it is well to filter off the antipyrin precipitate as rapidly as possible. C. A. M.

Analysis and Composition of Liquorice Extract. G. Py. (*Journ. Pharm. Chim.*, 1897, 280-284.)—The presence of gelatin in liquorice may be detected by the precipitate obtained with tannin, phosphomolybdic acid, picric acid, etc., in an

aqueous solution of the portion insoluble in 75 per cent. of alcohol. (See abstract, this volume, p. 192.)

If only an examination for gelatin is required, the original sample may be tested in the following manner: About 5 grammes of the extract are dissolved in 50 c.c. of hot water, allowed to cool, and the solution saturated with ammonium sulphate to precipitate the glycyrrhizin and gelatin (if present). The precipitate is washed with a saturated solution of ammonium sulphate, and then with alcohol (75 to 80 per cent.). This dissolves the glycyrrhizin, and any precipitate left can be dissolved in water and tested for gelatin by the usual reactions.

Gum may be looked for in the liquid which has been tested for gelatin (*i.e.*, aqueous solution of substances insoluble in 75 per cent. alcohol). (1) It gives a white precipitate when tested with a mixture of three drops of a solution of sulphate of copper (1 : 10) and 10 c.c. of potash (45° Bé). (2) It gives a precipitate with ferric chloride to which an equivalent of calcium carbonate has been added. These tests give negative results with pure liquorice extract.

A microscopical examination will detect the presence of starch and lamp-black, which are frequently found in liquorice pastilles.

The following table gives the results of the analyses made by the author:

	Pure Extract.	Pharmaceutical Extracts.		Commercial Extracts.													
Per cent.	1.	2.	3.	4.	5.	6.	7.	8.	8*a*.	9.	10.	10*a*.	10*b*.	10*c*.	10*d*.	10*e*.	11.	11*a*.
Water	11·52	12·46	8·82	7·94	9·98	10·18	5·16	8·86	16·00	9·40	11·14	13·62	12·72	15·46	7·58	13·50	9·88	10·80
Soluble ash	3·22	1·92	2·24	1·76	2·36	2·78	0·40	3·60	3·54	2·46	3·48	4·36	4·06	4·30	4·38	4·56	1·92	2·18
Insoluble ash	2·46	2·70	1·76	2·02	3·98	4·02	1·52	2·76	2·38	2·00	2·64	2·86	2·52	2·70	2·84	2·76	1·80	1·90
Total ash	5·68	4·62	4·00	3·78	6·34	6·80	1·92	6·36	5·92	4·46	6·12	6·72	6·58	7·00	7·22	7·32	3·72	4·08
Alcoholic extract	72·70	47·46	44·10	54·15	53·15	48·50	37·60	50 05	47·62	47·05	50·00	49·25	49·16	44·35	51·45	48·15	49·80	47·10
Organic substance insoluble in 70 per cent. alcohol	10·10	35·46	43·08	84·15	30·53	34·52	55·82	34·73	30·48	39·09	32·74	30·41	31·54	33·19	33·75	31·03	36·06	38·02
Ammonium glycyrrhizate	14·96	5·76	9·90	11·90	13·03	11·00	9·70	13·10	8·00	7·15	13·34	9·80	6·68	5·80	12·05	11·40	4·02	6·25
Reducing sugars	18·84	14·28	8·00	13·50	12·60	7·40	13·50	10·00	..	14·00	..	..	12·04	..	7·40	..	..	..
Total nitrogen	2·08	2·81	5·20	1·98	1·96	1·90	..	..	..	..	..	..	5·36	..	4·83	..	..	..
Gelatin	—	—	+	—	—	—	..	+	+	+	+	+	+	+	+	+	—	—
Microscopical examination.	Liquorice starch.	Liquorice and Potato starch.	Farina and Liquorice starch.	Liquorice small amount.	Rice.	Foreign starches.	Farina large proportions.	Small amount of rice.		Farina.	Liquorice starch granules, occasionally granular albuminoid matter.						Considerable amount of foreign starches.	

The pure liquorice extract was prepared in the laboratory, while the others were representatives of the ten brands most commonly occurring in the market. With the exception of number seven, which consisted of a mixture of starch, sugar, glycyrrhizin, and gum, the extracts, whether containing gelatin or not, only showed a marked difference in the amount of glycyrrhizin. In the author's opinion all extracts containing less than 10 per cent. of that constituent must be regarded as derived from spent liquorice.

C. A. M.

ORGANIC ANALYSIS.

Determination of Aldehyde in Ether. M. Francois. (*Jour. Pharm. Chim.*, 1897, v., 521-525.)—The proposed method consists in the application of Schiff's reagent as a colorimetric quantitative reagent. The colour produced by the sample is compared with those yielded by ether containing known quantities of aldehyde.

C. A. M.

On the Estimation of Formaldehyde. G. Romijn. (*Zeit. anal. Chem.*, 1897, xxxvi., 18-24.)—The methods of Legler, Brochet, and Cambier were studied, and the results obtained with them compared with those given by two new methods described by the author. For this purpose four aqueous solutions were prepared containing in 500 c.c.: (1) 2·075 grammes of formalin; (2) 2·075 grammes of formalin + 1·3 gramme of acetaldehyde; (3) 2·075 grammes of formalin + 0·355 gramme of acetone; (4) 2·075 grammes of formalin + 1 gramme of benzaldehyde.

Iodometric Method.—Ten c.c. of the aldehyde solution are mixed with 25 c.c. of decinormal iodine solution, and sodium hydrate added drop by drop until the liquid becomes clear yellow. After ten minutes hydrochloric acid is added to liberate the uncombined iodine, which is then titrated back with standard thiosulphate. Two atoms of iodine are equivalent to 1 molecule of formaldehyde. The results obtained with the first solution showed that the formalin used contained (1) 37·38 and (2) 37·40 per cent. of formaldehyde.

With the second solution a certain amount of iodoform was produced, and the results were too low. With the third solution the acetone was entirely converted to iodoform, and in the fourth solution the benzaldehyde was partially oxidized. Hence this method, though suitable for the valuation of pure formaldehyde, does not give correct results in the presence of other aldehydes.

Potassium Cyanide Method.—This is based on the fact that formaldehyde combines with potassium cyanide. The addition product reduces silver nitrate in the cold. But if the silver nitrate be acidified with nitric acid before the addition of the aldehyde cyanide mixture, no precipitate results if the aldehyde in the latter be in excess. If, on the other hand, the potassium cyanide is in excess, one molecule of potassium cyanide is left in combination with one molecule of the formaldehyde, while the excess precipitates silver cyanide from the silver nitrate solution.

Ten c.c. of decinormal silver nitrate, acidified with nitric acid, are mixed with 10 c.c. of potassium cyanide solution (prepared by dissolving 3·1 grammes of the 96 per cent. salt in 500 c.c.), the whole diluted to 500 c.c., filtered, and 25 c.c. of the filtrate titrated by Volhard's method. The difference between this blank result and that obtained by titrating the filtrate after the addition of the aldehyde solution gives the amount of standard sulpho-cyanide corresponding to the silver not precipitated by the excess of potassium cyanide. From this the amount of formaldehyde can be calculated. With solution 1 the results showed 37·39 and 37·67 per cent. of formaldehyde in the formalin. With solution 2, if the titration was made immediately after shaking, only the formaldehyde had combined, but if left for some time the acetaldehyde also began to combine, and erroneous results were obtained. Solutions 3 and 4 gave correct results, even after standing for thirty minutes.

Hydroxylamine Method (Brochet and Cambier, *Comp. Rend.*, cxx., 449).—This gave satisfactory results with pure formaldehyde, but quite irregular figures with the other three solutions.

Legler's Method (Ber., xvi., 1335).—The four solutions were made more concentrated, in order to lessen the difficulty of observing the end reaction. In each case the correct amount of formaldehyde was found, but the author does not consider the method so accurate as the others. C. A. M.

On the Low Iodine Number of Linseed-Cake Oil. G. Fassbender and J. Kern. (*Zeit. angew. Chem.*, 1897, 331, 332.)—Ketel and Antusch (ANALYST, xxi., 299) came to the conclusion that the low iodine value of oil extracted from linseed-cake was due to the presence of cotton-seed oil, and not to foreign seeds in the original linseed. The authors show that this conclusion may be incorrect, for on pressing in the cold linseed, containing a small percentage of the usual foreign seeds, a separation of the oils takes place, the linseed-oil flowing away, whilst the heavier crucifer oils remain behind; so that linseed-cake prepared from impure linseed contains a much more impure linseed-oil than does the original seed. This is shown by the iodine values given in the following table:

Oil from	Pure Linseed.	Impure Linseed, containing 5·3 per cent. Crucifer Seeds.
I. Cold-pressed. Per cent. of oil brought from 37 to about 25	180·5	180·5
II. Pressed at 70°. Per cent. of oil brought from 25 to 12	180·3	175·3
III. Extracted. From 12 per cent. to 0 ...	179·9	162·5

C. A. M.

Krüger and Wulff's Process for estimating Alloxuric Substances (Xanthine Bases) in Urine. Huppert (*Zeits. Physiol. Chem.*, 1897, xxii., 556); also **Salkowski** (*D. Med. Wochenschr.*, 1897, xxiv., 14; through *Chem. Zeit. Rep.*, 1897, 105.)—Both authors state that this process is not reliable, for the mixture of copper sulphate and sodium bisulphite used by Krüger and Wulff yields a precipitate containing too much nitrogen when it is calculated into alloxuric substances, this being due to the fact that the reagent throws down not only xanthine bases and uric acid, but also albumin, secondary albumoses, and thiocyanogen (according to Huppert) when these bodies are present in the urine. F. H. L.

INORGANIC ANALYSIS.

The Determination of Sulphur in Pyrites and Blende. M. Noaillon. (*Bull. de l'Ass. belge*, 1897, x., 443-444.)—A weighed quantity (1·5 gramme) of the sample is oxidized with 10 c.c. of nitric acid and 10 c.c. of a 30 per cent. solution of sodium chlorate, the object of the latter being to provide a base to combine with the free sulphuric acid liberated during the reaction. The flask is placed in water to keep the contents cool, and the oxidation is complete in thirty minutes, without separation of sulphur, and almost without liberation of gas.

The nitric acid is then expelled by evaporating twice at 200° with hydrochloric acid. The residue is dissolved in a little hydrochloric acid, the solution diluted, and the iron precipitated with ammonia.

The cooled liquid is made up to 600 c.c., and filtered, without washing the precipitate. 200 c.c. of the clear filtrate (=0·50 gramme pyrites) are boiled to expel excess of ammonia, acidified, and the sulphuric acid determined in the usual manner by precipitation with barium chloride.

The influence of the volume of precipitated ferric hydrate is quite negligible in a solution of 600 c.c.

Although, if the blende be pure, there is no liberation of acid, as in the case of pyrites, it is always advisable to introduce a base as above, since commercial blendes frequently contain more or less pyrites. C. A. M.

The Use of Acetylene in Quantitative Analysis. H. G. Söderbaum. (*Ber.*, 1897, xxx., 902-904.)—Acetylene precipitates copper from ammoniacal solutions of the latter as black copper-acetylide. The reaction is complete, and no trace of copper can be detected in the filtrate after the acetylide has been filtered off. The following mode of carrying out the estimation of copper by means of acetylide is recommended: The salt is dissolved in 100 to 200 times its weight of water contained in an Erlenmeyer flask, made ammoniacal and gently warmed on the water-bath. Acetylene is then passed through to saturation. Although the precipitation is complete in the cold, it is better to warm the solution, in order to hasten the reaction and to make the precipitate more flocculent. After filtering off and washing, the acetylide is digested for half an hour with warm dilute nitric acid. The black residue is filtered off, and the filtrate evaporated and gently ignited, when copper oxide is obtained. The filter and residue are separately burned, and the small amount of oxide added to the first quantity of copper oxide. Accurate results can be obtained.

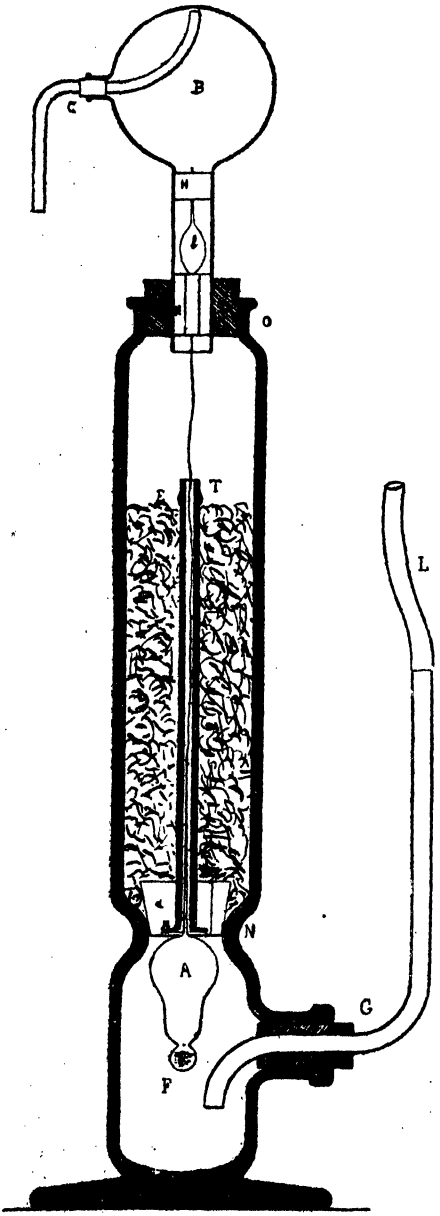
The presence of alkaline salts does not influence the precipitation, whilst it is retarded by ammonium salts.

The Separation of Copper from Zinc.—The presence of zinc salts prevents the precipitation of copper acetylide. If, however, a little sulphurous acid is added to the solution before passing in the acetylene, the copper is thrown down as a reddish-brown precipitate, which gradually becomes black. It may be filtered off and treated as above. The results, as shown by a number of figures given in the paper, are quite reliable. W. P. S.

APPARATUS.

Automatic Generator for Sulphuretted Hydrogen. Crouthière and Désolu. (*Ann. de Chimie Analyt.*, vol. ii. [2], 21-24.)—To prevent the overflow of the generating liquid into the receiver when the pressure of the gas no longer suffices to maintain it in equilibrium, the authors have designed an apparatus with automatic valves. The generator consists of a vertical calcium chloride tube connected with the reservoir of acid by the lower tubulus and attached tubing. A long 8 m.m. bore tube, flanged at its lower end, is passed through a cork fluted at the sides, which rests on the constriction in the generator. This tube serves as a guide for the stem of a hollow glass float loaded with 20 grammes of mercury and placed in the lower part of the generator, the upper part being then charged with ferrous sulphide nearly up to the top of the narrow tube. In the caoutchouc stopper at the mouth of the generator

is fitted a reversed half-litre bulb tube, the neck of which contains a glass bulb drawn out at either end to a point; these ends rest in guide tubes, and pass through corks at the top and bottom of the neck, the lower cork being fluted round the sides. The lower end of this bulb-float is connected with the stem of the lower float by a platinum wire. The bulb is pierced at one side by a tubulus, through which passes a tube leading to the gas-washing apparatus, the tube itself being turned up inside the bulb to prevent splashing over when the reaction is violent. The acid, on being admitted into the generator, rises through the channels in the cork, reacts on the ferrous sulphide, and ascends the tube, as the pressure of gas diminishes, until it reaches the neck of the bulb, where it raises the second float, and by closing the upper tube is prevented from ascending further. The gradual evolution of gas then forces the liquid down once more until the lower float is passed, and this, in descending, draws with it the upper float and re-opens the upper valve, thus relieving the pressure and allowing a fresh quantity of acid liquor to enter from the reservoir (the spent portion falling to the bottom on account of its greater density), the operation being repeated automatically until the acid is exhausted. The mixture of the fresh and spent liquids in the reservoir is effected by adjusting the bulb-tube so that the lower float does not draw the upper one until the liquid has sunk to the level of the feed-tube, whereby the gas bubbles up through the latter into the reservoir for a moment.



In unmounting the apparatus, the lower bulb is floated in liquid so as to release the tension on the wire, which is then unfastened, after removing the inverted bulb-flask, the tube and charge being withdrawn by a running-noose cord, which catches on a bulge at the top of the tube. All the corks are steeped in paraffin to preserve them from the action of the acid, and the reaction of the sulphuretted hydrogen on any free chlorine present prevents the latter from attacking the platinum wire.

C. S.

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NOTE ON THE INDIRECT (TABARIE'S) METHOD FOR THE ESTIMATION OF ALCOHOL.

By Norman Leonard, B.Sc., F.I.C., and Harry M. Smith, F.I.C.

(*Read at the Meeting, June* 2, 1897.)

In this well-known process the amount of alcohol in a spirituous liquid is calculated from two data: (1) the specific gravity of the original liquid, (2) the specific gravity of the liquid after removal of alcohol by evaporation and subsequent making up to the original volume with water. In the method as first published the specific gravity of the alcohol evaporated was stated to be found by dividing the specific gravity of the original liquid by that of the de-alcoholized liquid. For some time, however, a difference of opinion has existed as to the proper method of calculation, and, instead of the original "division" formula, a "subtraction" formula has been frequently employed. In the latter case the specific gravity of the alcohol evaporated is obtained by subtracting from the specific gravity of the original liquid the excess over unity of the specific gravity of the de-alcoholized residue. This calculation has been claimed to be both simpler and more in accordance with experimental results than the "division" formula. A few experiments made with a view to decide the relative value of the two methods have been recorded by Allen, Hehner, and Blunt (Analyst, xvi., 221, 223; xvii., 5), but no systematic investigation of the subject appears to have been carried out. The following observations and experiments are put forward in the hope of throwing some light on the matter.

Looking at the matter from the simple theoretical standpoint, apart from any errors involved in the actual working of the process, we have, for comparison, the specific gravities of three liquids:

1. An aqueous solution containing per 100 c.c., say, p. grammes of alcohol and q. grammes of non-volatile dissolved matter. The specific gravity of this "original" liquid we denote by O.

2. A solution containing per 100 c.c. p. grammes of alcohol, but no other dissolved substances. Let the specific gravity of this "distillate" be denoted by D.

3. A solution containing per 100 c.c. q. grammes of dissolved solid matter, but no alcohol. The specific gravity of this "residue" is denoted by R.

According to the division formula $D=\frac{O}{R}$, and by the subtraction formula $D=O-R+1$; the two equations may be written $\frac{1-D}{1}=\frac{R-O}{R}$ and $1-D=R-O$ respectively. Thus the subtraction formula is based on the assumption that the lowering of density due to the presence of alcohol is the same whether dissolved solid matter be present or not. The division formula, on the other hand, assumes that this lowering is proportional to the density of the de-alcoholized liquid. Or, in other words, the subtraction formula assumes that the substances in solution exert their influence on the density of the liquid, irrespective of each other's presence. The division formula, however, implies an expansion increasing in amount both with the amount of alcohol and with the amount of extractives.

It should be observed that the division formula must necessarily give a higher specific gravity, corresponding to a lower percentage of alcohol, than the subtraction formula. For the difference between the two values obtained for D, viz., $\frac{O}{R}$ and $O-R+1$ is $\frac{(R-O)(R-1)}{R}$, and this is always positive. The difference increases both with the amount of alcohol and with the amount of dissolved solids.

In the accompanying table are given the results of a number of experiments which were carried out in the following manner. Mixtures of pure cane-sugar, alcohol of known specific gravity, and water were made, the substances being carefully weighed out in stoppered flasks. After mixing, the solutions were allowed to stand some hours, and the specific gravity determined. The percentage composition by weight and by volume of each solution could now be calculated. The figures are given in the first four columns of the table. Having previously made experiments on the specific gravity of solutions of cane-sugar, we were able to calculate what the specific gravity of each solution would be if deprived of alcohol. This figure is given in column 6. Similarly, from the usual alcohol tables, we found the specific gravity of a liquid containing the alcohol actually present, but devoid of sugar (column 7). Thus, in the first experiment each 100 c.c. of the solution contained 1·950 grammes of sugar and 2·201 c.c. of alcohol. Its specific gravity was 1·0043. The specific gravity of a 2·201 per cent. by volume solution of alcohol is 0·9969, while the specific gravity of a solution of cane-sugar containing 1·950 grammes per 100 c.c. is 1·0076. These are therefore the calculated specific gravities of the "distillate" and of the "residue" respectively. In columns 8 and 9 are given the results of the application of the subtraction formula to the numbers in columns 5 and 6, together with the differences between these results and the numbers in column 7. In columns 10 and 11 are given similar figures for the division formula.

The results of the first series of experiments, in which the sugar amounted throughout to about 2 per cent., show that the division formula gives accurate results for all strengths of alcohol. The subtraction formula, on the other hand, always gives low results, the deviation steadily increasing with the amount of alcohol present.

In the second and third series, with 10 and 27 per cent. of sugar respectively, the same steady increase in the deviation of the subtraction formula is seen, but the division formula is no longer trustworthy.

We are unable to detect any simple relation between the numbers we have obtained, and, taking into consideration the fact that so little is known, except empirically, of the connection between the density and composition of even simple solutions, it is not to be expected that a simple and accurate expression of the facts should be found in the more complicated case of two substances in solution. In the practical application of Tabarie's process, moreover, we are met with further difficulties in the presence of dissolved substances other than sugar and of volatile substances other than alcohol. Of the latter, some—*e.g.*, carbonic acid—may be present in the original liquid, but not in the distillate, thus introducing another source of error, as was pointed out by S. Harvey (ANALYST, xvii., 1).

Since writing the above, our attention has been called to a paper by H. Droop Richmond (*J. Fed. Inst. Brewing*, 1896, ii., 529-535), in which an attempt is made to theoretically demonstrate the validity of the subtraction formula. The reasoning, however, is based upon the assumption that the solution densities of alcohol and of the extractives are the same, whether only one or both substances are present in solution. As we have pointed out above, the subtraction formula does indeed imply that the substances exert their specific influence on the density of the solution, independently of each other's presence; but this supposition is not in accordance with the results of our experiments. The latter show that when solutions of sugar and alcohol are mixed, a slight expansion takes place, the actual density of the mixture being lower than that calculated by the subtraction formula.

Our thanks are due to Dr. Thomas Stevenson for permission to carry out these experiments in his laboratory.

SERIES I.

	1.	2.	3.	4.				8.	9.	10.	11.
	Per 100 grammes.		Per 100 c.c.					Subtraction Formula.		Division Formula.	
	Sugar (grms.).	Alcohol (grms.).	Sugar (grms.).	Alcohol (c.c.).	O.	R.	D.	O − R + 1.	Difference.	$\frac{O}{R}$	Difference.
1	1·944	1·740	1·950	2·201	1·0043	1·0076	0·9969	0·9967	− 0·0002	0·9967	− 0·0002
2	1·979	7·870	1·966	9·859	0·9944	1·0076	0·9870	0·9868	− 0·0002	0·9869	− 0·0001
3	1·992	16·218	1·957	20·093	0·9835	1·0076	0·9762	0·9759	− 0·0003	0·9761	− 0·0001
4	2·103	25·295	2·042	30·974	0·9720	1·0079	0·9645	0·9641	− 0·0004	0·9644	− 0·0001
5	2·142	42·940	2·012	50·847	0·9400	1·0078	0·9329	0·9322	− 0·0007	0·9327	− 0·0002
6	2·326	74·522	2·015	81·379	0·8668	1·0078	0·8601	0·8590	− 0·0011	0·8601	Nil.
7	2·403	84·414	2·021	89·535	0·8420	1·0078	0·8355	0·8342	− 0·0013	0·8355	Nil.
					SERIES II.						
	9·652	1·481	9·988	1·933	1·0359	1·0387	0·9972	0·9972	Nil.	0·9973	+ 0·0001
	9·843	4·458	10·139	5·790	1·0311	1·0392	0·9920	0·9919	− 0·0001	0·9922	+ 0·0002
10	9·767	7·649	10·017	9·882	1·0256	1·0388	0·9870	0·9868	− 0·0002	0·9873	+ 0·0003
11	11·753	17·207	11·975	22·106	1·0198	1·0463	0·9741	0·9735	− 0·0006	0·9747	+ 0·0006
12	9·986	20·654	10·056	26·225	1·0079	1·0389	0·9698	0·9690	− 0·0008	0·9702	+ 0·0004
13	10·432	27·724	10·400	34·852	0·9979	1·0403	0·9596	0·9576	− 0·0020	0·9592	− 0·0004
14	10·216	32·492	10·082	40·486	0·9879	1·0390	0·9515	0·9489	− 0·0026	0·9508	− 0·0007
15	10·985	34·541	10·822	42·910	0·9861	1·0419	0·9471	0·9442	− 0·0029	0·9464	− 0·0007
16	10·901	45·185	10·469	54·720	0·9613	1·0405	0·9249	0·9208	− 0·0041	0·9239	− 0·0010
					SERIES III.						
17	24·157	1·396	26·526	1·933	1·0991	1·1017	0·9972	0·9974	+ 0·0002	0·9976	+ 0·0004
18	25·211	7·174	27·524	9·877	1·0928	1·1055	0·9870	0·9873	+ 0·0003	0·9885	+ 0·0015
19	25·647	14·604	27·733	19·906	1·0820	1·1063	0·9764	0·9757	− 0·0007	0·9780	+ 0·0016
20	25·652	22·188	27·361	29·841	1·0676	1·1049	0·9658	0·9627	− 0·0031	0·9662	+ 0·0004

Discussion.

Mr. Hehner thought that the paper had a special value as giving experimental data which would be of use in arriving at a settlement of this question. He did not think that purely theoretical conclusions were of much use in themselves. A very much more extended series of experiments was to be desired, though these of Messrs. Leonard and Smith appeared to strongly favour the division formula.

Mr. A. C. Chapman remarked that, in the case of beer, if one subtracted the present gravity from the extract gravity, a number was obtained which was always slightly lower than the difference between the distillate gravity and unity. Judging from the numbers given by the authors, this did not appear to be the case with the solutions examined by them.

The President said that, as nearly as he could recollect, Mr. Chapman's observations were in accord with the experience of Mr. Harvey, whose work had been referred to in the paper. Despite all the work that was being done in physical chemistry, there was still much to learn about the state of bodies in aqueous solution. The aggregation of molecules and the degree of hydration might be very sensibly affected by dilution with alcohol in directions that might give rise to some of the apparent discrepancies that had been referred to.

NOTE ON THE DETERMINATION OF PROTEIDS BY CHLORINE.

By S. Rideal, D.Sc. Lond., and C. G. Stewart.

(*Read at the Meeting, June* 2, 1897.)

In 1876 one of us, in conjunction with the late Dr. Bernays, made a large number of experiments with the object of determining the presence and amount of gelatin in meat-extracts. In Liebig's original formula, gelatin and fat were intentionally excluded from such meat-extracts; consequently, one of the first questions asked of an analyst in examining a meat-extract was, "Does it contain any gelatin?" Inasmuch as the latter is obtained from the cheaper parts of meat, or from other sources, and gives to the extract a spurious solidity and "strength," it was regarded, and is still considered to be, an undesirable constituent, which under certain circumstances may amount to an adulteration. The determination of gelatin became, therefore, of considerable importance, and at the present time the method based upon its insolubility in ice-cold water has been apparently adopted as the last resource of the analyst. Experiments, in which an attempt was made to utilize the tannin reaction, either by weighing the precipitate, or by adding a slight excess of tannin solution and titrating back with permanganate, gave very unsatisfactory results. Other processes, based upon the use of ferrocyanide and on dialysis, also failed to give any reliable quantitative results.

The reaction with chlorine water in excess was subsequently tried, and the insolubility of the precipitate with this reagent suggested its use as a means of

determination. The precipitate subsides in compact flocks, is easily filtered, and yields a clear filtrate. By weighing the precipitate, after drying at a low temperature, results were obtained with gelatin which were comparable with the tannin process when worked under the most favourable conditions, but much more easily, and with a far better agreement between repeated analyses. The process was not published, but having had a number of meats and meat-extracts to examine last year, we decided to undertake a further investigation into it.

It was found that a current of chlorine precipitated in a weighable form all albuminous substances, that the precipitation was delicate and complete, and that the bases, such as creatine, etc., were not precipitated. The chlorine compound was remarkably stable at ordinary temperatures, but if heated it readily decomposed, becoming nearly black, and rotting the filter-paper. In the precipitation hypochlorous acid seemed to be the principal product. Finally, the following procedure was adopted :

A hundred c.c. of the liquid, containing not more than 0·2 per cent. of proteids, were submitted in a beaker to a steady current of chlorine. The phenomena noticed in every case were similar. The solution remained for a time clear, then it began to froth strongly, the bubbles being encased with a white film, and at this period it should be stirred with the delivery-tube. In a short time the frothing ceases, the precipitate becomes granular and the liquid clear. As soon as the latter shows a marked yellow colour the current of chlorine is stopped. The solutions can be filtered at once, although it is preferable to allow them to stand for some hours, as the precipitate then becomes more compact and more easily filtered. It is collected on a weighed Schleicher and Schüll's hardened filter-paper, 9 cm. diameter, used with a water-pump, washed thoroughly with cold water until all free chlorine is removed, and the paper and its contents drained and allowed to dry, as far as possible, in warm air, the drying being finished *in vacuo* over sulphuric acid. In the earlier experiments the precipitate when nearly free from water was finally dried in a water-bath ; but if the temperature exceeds 70° or 80° C. decomposition ensues, and the results are never so uniform as by drying *in vacuo*. The dried precipitate is of a pale, yellowish-white colour, pulverulent, and quite inodorous. In an earlier experiment to determine the relation of the weight of the chlorine precipitate to that of gelatin, a sample of the white commercial gelatin was dissolved to a 1 per cent. solution, and 25 c.c. of this solution diluted to 100 c.c. were then precipitated with chlorine. The product, dried at 70° to 80° C., weighed 0·213 gramme, so that 1 gramme of the gelatin used yielded 0·852 gramme of chlorine precipitate. As the sample was found to contain 25·53 per cent. of water, it followed that 0·852 gramme of chlorine precipitate was yielded by 0·7447 gramme of anhydrous substance (not ash-free). This determination gave the factor 0·87 for multiplying the weight of the chlorine precipitate in order to convert it into the equivalent of dry commercial gelatin.

To further examine this factor, a solution of " Coignet's Extra Gelatin," of about 9 per cent. strength, as prepared for bacteriological work, but with exclusion of the other nitrogenous matter, was analysed by the several methods. The original material contained 19·27 per cent. water and 2·96 per cent. ash, leaving 77·7 per cent. of organic matter, or approximately 7 per cent. in the solution.

The results of a duplicate analysis were as follows:

	Grammes per 100 c.c. I.	II.
Total nitrogen	1·242	1·246
× 5·5 = gelatin	6·83	6·86
Nitrogen in ammonium sulphate precipitate ...	1·109	—
× 5·5 = ...	6·10	—
Nitrogen in Stutzer precipitate	0·514	0·528
× 5·5	2·83	2·90
Stutzer filtrate precipitate by ammonium sulphate (direct weighing)	4·37	4·27
Total proteid	7·20	7·17
Chlorine precipitate	9·375	9·300
× 0·87	8·19	8·12
× 0·74	6·94	6·88

These results show that the factor 0·87, which was deduced from the first experiment, making no allowance for ash, gives too high a result. Taking the total nitrogen × 5·5 as representing the total proteid present, the factor 0·74 gives the actual relation of the chlorine precipitate to the dry and ash-free proteid in this gelatin solution. We were surprised to find that Stutzer's reagent, made according to the directions of Heaton and Vasey (ANALYST, 1892, p. 29), gave a precipitate with the solution, as the gelatin used appeared to be pure and of the best quality, and was analysed in a perfectly fresh and sterilized condition. This solution was prepared for studying the bacteriolysis of gelatin to be referred to in a subsequent paper.

It seems probable that the Stutzer reagent precipitates a portion of the gelatin, which may be partly hydrolysed in its manufacture. We have, however, noticed from several experiments that the most consistent jellies give the largest precipitate with Stutzer's reagent. The Stutzer precipitate from a gelatin, when decomposed by sulphuretted hydrogen, yields a jelly, so that we are inclined to the belief that it cannot be looked upon as a reliable reagent for differentiating between gelatin and albumose in a solution consisting mainly of gelatin. The following figures, obtained from three gelatins, "somatose," and Witte's peptone, will illustrate this point, and also the mean factor, 0·78, with which it is proposed to multiply the chlorine precipitate to attain the total proteids:

	Nelson's No. 1 Gelatin.	Coignet's Extra Gelatin.	Swiss Gold Label Gelatin.	Somatose.	Witte's Peptone.
Water	18·96	17·85	17·84	7·3	7·62
Ash	4·19	1·5	1·47	1·06	2·46
Total nitrogen	13·8	14·61	14·33	13·72	14·67
× 5·5	75·9	80·35	78·81	—	—
× 6⅓	—	—	—	86·75	92·91
Total organic matter ...	76·85	80·65	80·69	91·64	89·92
Nitrogen in ammonium sulphate precipitate	13·69	13·63	13·92	12·44	10·13
Nitrogen in phospho-tungstic precipitate	13·72	14·00	14·16	12·65	12·65

	Nelson's No. 1 Gelatin.	Coignet's Extra Gelatin.	Swiss Gold Label Gelatin.	Somatose.	Witte's Peptone.
Nitrogen in Stutzer precipitate	4·09	5·91	4·59	6·64	4·52
Chlorine precipitate ...	99·2	103·48	97·8	103·6	92·0
× ·78	77·38	80·60	76·28	80·81	71·76
Nitrogen in phosphotungstic precipitate :					
× 5·5	75·46	77·0	77·88	—	—
× 6·3	—	—	—	79·67	79·67

It will be noticed from the above results that the total nitrogen in the three gelatins gives the factors 5·56, 5·52; 5·63 for conversion into total organic matter present, and these figures are in fair accord with the usual factor 5·5 for gelatin. The gelatin, calculated from either the nitrogen in the ammonium sulphate precipitate or the phosphotungstic acid precipitate, would give slightly lower results, due to either incomplete precipitation or losses in the two processes. The Stutzer reagent (perfectly neutral) gave, as in former experiments, a precipitate with all three gelatins, and duplicates with a fresh reagent confirmed these determinations, but as the second preparation was slightly alkaline the results were somewhat higher.

Although the solutions were of equivalent strength, and the analysis shows that gelatins 2 and 3 had approximately the same amount of water and ash, gelatine 2 gave a very much more consistent jelly than gelatin 3. This discrepancy of physical character manifests itself in the lower Stutzer and chlorine figures in No. 3, while the ammonium sulphate and phosphotungstic results show a tendency in the opposite direction.

In the somatose and peptone, any calculation from the total nitrogen gives results which do not represent the total proteids present. The ammonium sulphate and phosphotungstic acid give very close agreement in the case of somatose, showing the absence of peptone.

The chlorine precipitate figures, when multiplied by 0·78, give results which seem in close accord with the known amount of proteid present in all cases except the peptone. The amount, however, is higher than that given by the ammonium sulphate determination, so that peptone is precipitated by chlorine, but the weight of the precipitate is probably less than that of the other proteids. Assuming 6·3 as the nitrogen equivalent of the proteids in Witte's peptone, and that phosphotungstic acid precipitates all the proteid matter, but none of the bases, we have 79·67 per cent. of total proteids, as against 71·76 per cent. found by chlorine on the basis of the factor 0·78.

The chlorine precipitates from different sources show a remarkable similarity. The amount of nitrogen in a number of them was found by Kjeldahl's process. The results were :

In Precipitates dried finally at 70° *to* 80° *C.*—Meat-extracts (containing albumoses, gelatin, and peptones), three different samples, 13·91, 13·79, and 14·27 per cent. of nitrogen.

In Precipitates dried in a bell-jar over Sulphuric Acid.—Two samples of meat-extract, 12·00 and 12·24. A gelatin, 11·99. Egg-albumen, 12·15. Another gelatin, 12·75.

In Precipitates also dried in vacuo over Sulphuric Acid.—Swiss gold-label

gelatin, 13·88. Nelson's No. I., 13·37. Coignet's extra gelatin, 14·09. Somatose, 13·40. Witte's Peptone, 13·53.

It will be noticed that the average of these five last determinations, 13·65, is exactly three-quarters of the percentage in gelatin, 18·2, on which the factor 5·5 is founded. The nitrogen in the chlorine precipitate multiplied by 7 3 gives in these cases the weight of crude proteid.

The process had been tried on a number of meat-extracts, prepared in the laboratory according to Liebig's method by digesting 1 lb. of meat with 1 pint of water for six hours, heating to coagulate albumen, etc., and filtering. The chlorine precipitates, calculated by the factor first used, 0·87, gave percentages of soluble proteids amounting in the five cases to 1·83, 1·65, 1·92, 1·98, and 1·51, the results agreeing with the detailed analyses in assigning their relative values to the products.

At the time of the inception of the investigation, it was found that bromine also precipitated the proteids; an attempt was therefore made to volumetrically estimate them by adding a known quantity of bromine in excess, and then titrating back with thiosulphate. The method was found to succeed fairly well in solutions of proteids free from bases, but inasmuch as the latter, though not precipitated, absorb much chlorine or bromine, the method was inapplicable to meat-extracts and to most organic fluids.

We are at present studying to produce a weighable precipitate by bromine instead of by chlorine, as the former reagent presents several advantages.

It is doubtful whether phosphotungstic and other precipitants do not also throw down from animal solutions some of the nitrogenous extractive acids and other substances which have not been sufficiently considered; chlorine, on the other hand, seems only to precipitate the proteids.

A careful search through chemical literature, which was made while these experiments were pending, yielded no record of the properties or composition of the chlorine precipitate; it was only incidentally mentioned. But more recently, in going back as far as 1840, it was found that this precipitate had actually been made use of by Mulder as one of the foundations of his protein theory. He called the precipitate "proteinchlorous acid" (*Berzel. Jahresb.*, xix., 734; *J. f. Chem.*, xliv., 489), and stated that there was first formed "chlorite of albumen, which produces sulphide of silver with silver salts, afterwards proteinchlorous acid, the sulphur of which no longer acts on silver (being probably oxidized). The acid is obtained of constant composition by drying at 80° C." He describes its properties in terms which are practically identical with our observations. The following early analyses are recorded:

COMPOSITION OF CHLORINE COMPOUND FROM THE SOURCES MENTIONED.

	MULDER.			VAN LAER.	
	Gelatin.	Albumen.	Fibrin.	Casein.	Hair.
C	46·66 46·25	48·0	48·2	48·48	48·40
N	15·59	14·11			14·09
H	5·90 5·81	6·21	6·16	6·38	6·08
O	23·37				
ClO_3	8·48 8·47	11·44	11·46	12·27	12·61

De Vrij (*Ann. Pharm.*, lxi., 248) and others denied the presence of chlorous acid, and a considerable controversy ensued as to the nature of the compound, although all agreed as to its properties, its constancy, and its insolubility. With the collapse of the protein theory this early work seems to have been forgotten. We will hazard at present no theory as to the constitution of the body, beyond pointing out that the percentage of nitrogen found by the early observers agrees closely with the amount found by us in specimens dried, as directed by Mulder, at 80° C., and that the formation of hypochlorous acid was invariably observed in our experiments.

See also Thenard (*Mem. d'Arcueil*, ii., 38), Mulder (*Bullet. en Neerlande*, 1839, 153), and Berzelius (*Jahresb.*, xix., 729).

Discussion.

Mr. Allen suggested that by making a Kjeldahl determination on the chlorine precipitate, it might be possible to avoid the necessity for weighing the precipitate itself. He was much interested in the references made to the use of bromine. Those who were in the habit of using halogen reagents would certainly use bromine in preference to chlorine whenever it was possible to do so. He thought the authors were to be congratulated on having hit upon what promised to be a very useful method of throwing down proteids. He would like, however, to know exactly what bodies they included in the term "proteids." It was sometimes difficult to know where to draw the line in a definition of this kind. Thus, some chemists excluded peptones from, and others included peptones in, the class of proteids.

Mr. Hehner said he would have liked to see some data as to the application of the method to other albuminoids than gelatin; and also to know if albumin, albumose, and peptone respectively were precipitated, and to what extent.

Mr. Richmond asked why the authors had made use of the particular factor 6·55. Was it obtained as the result of actual determinations of the mixture of proteids in the substance? It seemed to him extremely probable that all the gelatin might not be thrown down. It was shown that the precipitate, dried over sulphuric acid, contained 12 per cent. of nitrogen, and that the weight of this precipitate, multiplied by 0·87, gave the original gelatin. Now, 12 divided by 0·87 gave something under 14, and surely gelatin contained much more nitrogen than that. It might be that the Kjeldahl process was not reliable for a substance containing chlorine in organic combination, as free chlorine might be liberated, and this would act on the ammonia, and, neglecting intermediate products, produce free nitrogen. Nitrogen might be lost in this way. He would like to ask how the ammonium sulphate precipitates were determined, whether by actual weighing, or by determination of the nitrogen; and, if the latter, whether the factor 6·55 was used.

Mr. Bodmer said he understood that this process was intended for the estimation of gelatin, but as far as he could make out only the total proportion of gelatin and albumoses together appeared to be given. The determination of gelatin as such was really the most difficult point in the analysis of meat-extract.

Mr. Bevan inquired whether the nitrogen had been estimated in the filtrate from the chlorine precipitate. If so, this would answer the remark of Mr. Richmond as to whether the whole of the gelatin had been precipitated.

The PRESIDENT said that he had determined the nitrogen in one or two organic compounds containing chlorine or bromine, including potassium parabromochloronitrophenol, and had found no difficulty whatever. He could quite understand that the presence of chlorine might possibly give rise to trouble if it were to act as an oxidizing agent, because the permanganate of potash, which was in earlier times used in the Kjeldahl process, had a tendency to produce low results, owing to oxidation of the ammonia. He thought, however, that if there were any such tendency on the part of chlorine in organic combination, it might be got over by the use of zinc.

Mr. ALLEN said that one atom of chlorine could not combine with more than one atom of hydrogen, and as the number of hydrogen atoms was considerable, he did not think there was much chance of the chlorine acting as an oxidizing agent. Although the question was theoretically a very interesting one, he did not think it likely that it would assume a serious aspect in practice.

Dr. RIDEAL, in reply to Mr. Allen, said that he used the term "proteid" as an equivalent to "albuminoid," as he preferred the former to the latter when speaking of gelatin and gelatin derivatives. He would not venture on a definition, but proteids, he thought, ought not to be dialyzable; this would exclude peptones, which differed from the other proteids chemically, and the value of which as foods was still under discussion.

The following table shows the results obtained by kjeldahling the dry chlorine precipitate, from which it appears that chlorine carried down practically all the proteid nitrogen. There could be no doubt that the precipitation of gelatin and of other proteids by chlorine was complete, the only question being as to the composition of the precipitate.

No.	Description.	Weight of Proteids anhydrous and ash-free.	Weight of Chlorine precipitate.	Ratio of Chlorine precipitates to Proteids.	Kjeldahl Nitrogen in Proteid.	Kjeldahl Nitrogen in Chlorine precipitates.	Percentage of Nitrogen in Chlorine precipitates.	Ratio of Nitrogen in Chlorine precipitates to weight of Proteids.
	Coignet's gelatin	0·1160	0·158	0·734	0·0211	0·0218	13·8	5·32
	,, ,,	0·1084	0·147	0·724	0·0197	0·0210	14·28	5·16
3	Bovril fluid beef...	0·1951	0·2438	0·80	0·03147	0·03164	12·98	6·17
	Liebig Co.'s extract	0·0983	0·1130	0·87	—	0·01526	13·50	6·44
	Meat extract 2 S.	0·0499	0·0640	0·78	0·0079	0·0076	11·88	6·60
6.	Liquefied gelatin (*Micrococcus prodigiosus*)	0·1427	0·164	0·87	0·0219	0·0228	13·90	6·26
7.	Nelson's No. 1 gelatin	0·1813	0·2325	0·78	0·0326	0·0311	13·37	5 83
8.	Coignet's extra gelatin	0·1844	0·2365	0·78	0·0334	0·03234	14·09	5·70
9.	Swiss gold label gelatin ...	0·1821	0·2335	0·78	0·0327	0·0336	14·39	5·42
10.	Somatose...	0·2020	0·2590	0·78	0·0337	0·03472	13·40	5·85
11.	Witte's peptone	0·1794	0·2300	0·78	0·0285	0·0311	13·53	5·76

In reply to Mr. Richmond, he had given in the paper results calculated to the factor 5·5 for the nitrogen in gelatin, and 6·33 for other proteids. The factor 6·55 was used to show the agreement in the first series of experiments with the nitrogen results. The precipitate, when dried over sulphuric acid, contained from 13·5 to 14 per cent. of nitrogen, whatever its source might be. In precipitating with ammonium sulphate, they had found it best to estimate the nitrogen in the precipitate, as this gave better results than direct weighing. In reply to the President and Mr. Allen, kjeldahling the moist precipitate had not been tried, as they thought that loss of nitrogen might be occasioned. When the precipitate was dry, as shown in the above table, the whole of the nitrogen appeared as ammonia.

ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

FOOD AND DRUGS ANALYSIS.

Direct Detection of Nitrites in Milk. E. Riegler. (*Pharm. Central-H.*, 1897, xxxviii., 223; through *Chem. Zeit. Rep.*, 1897, 117.)—Normal milk contains neither nitrites nor nitrates. To detect nitrites, 20 c.c. of the sample are shaken with 0·05 gramme of Riegler's reagent (equal parts of naphthionic acid and β-naphthol) and 5 drops of strong sulphuric acid for a minute. One or 2 c.c. of strong ammonia are then added, and the whole again agitated. The existence of 0·001 gramme of N_2O_3 in 100 c.c. of milk produces an intense red colour, and 0·0002 gramme gives a pale rose. F. H. L.

Detection of Sesame Oil in Butter and Margarine colored with Curcuma. M. Hanause. (*Jour. Pharm. Chim.*, 1897, 350-351.)—The red colour given by curcuma with hydrochloric acid often causes uncertainty in the test for sesame oil. Finding that glacial acetic acid either prevents or considerably weakens the action of the acid on curcuma, the author has modified the test in the following manner: The melted butter is mixed with 5 c.c. of glacial acetic acid in a test-tube, which is heated until the acid boils. Three c.c. of hydrochloric acid containing furfurol are then added (see ANALYST, xix., 47, and xxi., 135), the tube well shaken, and allowed to stand. If the butter is free from sesame oil the acid has only a faint rose-violet tint; whereas, when the oil is present, the colour is reddish-violet, and the fatty layer above is a yellowish-rose when hot, and a yellowish-white when cold. If after twenty-four hours the tubes are placed horizontally on white paper, the acid which has acted on the butter not containing sesame oil shows a reddish-brown tint, whilst that in the other tube has a grayish-black shade. These reactions have been obtained with samples of butter and margarine containing 1 per cent. of sesame-oil.

C. A. M.

Preliminary Examination of Cheese. A. Forster and R. Riechelmann. (*Zeit. für öffent. Chemie*, iii., 159.)—In the course of the administration of German law by the police, it frequently happens that cheeses are sent to the analyst in large

batches at a time, and have to be examined as quickly as possible. In accordance with the agreed official methods of Saxony, these samples have first to be subjected to a preliminary examination, consisting, among other things, of the refractometric examination of the fat.

The authors recommend the following plan for the separation of the fat, which they claim possesses advantages over the methods of Henzold and Hefelmann (ANALYST, this vol., p. 159) in requiring but 3 to 5 grammes of cheese, and in being exceedingly quickly carried out. A portion of the cheese is cut up into pieces of the size of matches, and 3 to 5 grammes introduced into the lower and wide part of a Gerber's butyrometer, with both ends open. The lower opening is then closed with a caoutchouc stopper, and 6·5 c.c. of boiling water introduced. After shaking, about 6·5 c.c. of sulphuric acid, of 1·820 to 1·825 specific gravity, as recommended by Gerber, are introduced, and the whole shaken, to effect the solution of the cheese, which is usually complete in about a minute. The butyrometer is then filled up to the top of the graduated tube with hot water, and allowed to rest. The fat soon rises up the tube, but, if required, the separation can be accelerated by centrifuging. A drop of the fat is then removed and placed between the prisms of the refractometer.

The following table shows how small are the differences between the results obtained by the authors' method and the methods of von Raumer and Bremer:

Description of Cheese.	Differences in the Readings of the Refractometer.		
	Forster and Riechelmann.	von Raumer.	Bremer.
Swiss	−1·5	−1·5	—
Edam...	+0·3	+0·4	—
Cream	−1·7	−1·7	—
Gorgonzola	−1·6	−1·2	—
Camembert	−3·2	−3·2	—
Limburg	−2·1	−2·1	−2·4
Margarine-Romadour ...	+6·5	+6·9	+6·7
Brie	−2·0	−2·3	−2·3

H. H. B. S.

The Quantitative Determination of Carbohydrates in Food Stuffs. W. E. Stone. (*Jour. Amer. Chem. Soc.*, 1897, xix., 183-197, and 347-349.)—Most of the analyses of food material fail to give any information about the "nitrogen-free extract," in which are often grouped together several compounds, chiefly of a carbohydrate nature, which differ widely in their chemical and physical properties, and in their physiological action. The carbohydrates in vegetable food stuffs comprise sugars, starch in various modifications, substances closely associated with cellulose and designated as gums, pentosans, hemi-celluloses and oxy-celluloses, and, finally, cellulose itself. Reliable methods for the determination of each of these are already known, but, as a rule, they require the absence of the other substances. The method described by the author is based on the successive treatment of the same sample: (1) With boiling alcohol to remove sugars; (2) with cold water to remove dextrin and

soluble starch; (3) with diastase or malt infusion to remove starch; (4) with dilute boiling hydrochloric acid to convert gums, pentosans, etc., into soluble reducing sugars; (5) with boiling 1·25 per cent. sodium hydroxide, leaving behind the crude fibre.

From 50 to 100 grammes of the finely-powdered material are boiled with 500 c.c. of strong alcohol under a reflux condenser, or extracted with it in a Soxhlet apparatus. The alcoholic solution is filtered, and if it only contains sucrose, the amount of the latter is determined with the polarimeter. When more than one kind of sugar is present the alcohol is evaporated, the residue taken up in water made up to definite volume, and the sugars estimated by titration with Fehling's solution before and after inversion.

The residue from the alcoholic extraction may contain carbohydrates soluble in water, such as dextrin and soluble starch, especially when the materials have been subjected to the action of heat. This residue is therefore treated with 500 c.c. of water, and allowed to stand for eighteen to twenty-four hours with frequent agitation. The aqueous solution is then filtered through a linen filter, and when only dextrinous substances are present, concentrated, inverted, and titrated with Fehling's solution in the usual way. When soluble starch is present, as shown by iodine solution, the aqueous solution is concentrated to 200 c.c., and the total carbohydrates determined in an aliquot part. Another fraction is treated with excess of barium hydroxide, which precipitates the soluble starch, and the dextrin again determined in the filtrate by inversion and titration as before. The difference between this result and the preceding gives the amount of soluble starch in the fraction taken.

The residue from the aqueous extract is air-dried and weighed. Two grammes of it are boiled with 100 c.c. of water for thirty minutes, then cooled to 60° C., and mixed with 10 c.c. of an infusion made by steeping 10 grammes of finely-ground malt in 50 c.c. of water for two or three hours. The mixture is kept for two or three hours at a temperature not exceeding 65° C., and when the starch is completely converted, the solution is passed through a linen filter and the residue well washed with hot water. The filtrate is evaporated to 100 c.c., transferred to a 200 c.c. flask, mixed with 10 c.c. of hydrochloric acid, and kept in the water-bath for an hour. The solution is then neutralized, made up to 200 c.c., and titrated with Fehling's solution, a correction being made for the amount of sugar introduced in the malt-extract.

The residue from the malt extraction is mixed with 100 c.c. of water, and 2 c.c. of strong hydrochloric acid, and boiled under a reflux condenser for an hour. By this means the gums, pentosans, etc., are converted into reducing sugars, which may be regarded as xylose. The solution is filtered, neutralized, made up to 200 c.c., and titrated with Fehling's solution, each c.c. of which requires for complete reduction 0·004617 gramme of xylose. The equivalent of the xylose in xylan originally present may then be calculated according to the proportion :—

$$\underset{\text{Xylose}}{C_5H_{10}O_5} : \underset{\text{Xylan}}{C_5H_8O_4} :: 100 : x.$$

The residue from the hydrochloric acid treatment is boiled under a reflux condenser with 200 c.c. of 1·25 per cent. sodium hydroxide, filtered, washed, dried, weighed, ignited and weighed again, as in the manner prescribed for the estimation of

crude fibre by the Weender process. The substances extracted by the alkali are not hydrolysable to reducing sugars, and do not appear to be of a carbohydrate nature, though they often form a considerable proportion of the nitrogen-free extract.

The following table gives the percentages of different carbohydrates in certain food substances:

	Sucrose.	Invert Sugar.	Dextrin.	Soluble Starch.	Normal Starch.	Pentosans.	Cellulose.
Wheat I. ...	0·52	0·08	0·27	0·00	57·62	4·54	2·68
Wheat II. ...	0·72	0·00	0·41	0·00	56·27	4·37	2·51
Wheat flour I....	0·18	0·00	0·90	0·00	67·99	0·00	0·25
Wheat flour II.	0·20	0·00	1·06	0·00	67·76	0·00	0·25
Maize	0·27	0·00	0·32	0·00	65·45	5·14	1·99
Sugar beet (fresh) ...	8·38	0·07	0·35	0·00	0·00	4·89	1·00
Hay	2·53	0·00	1·18	0·00	7·49	7·18	34·34
Bread (whole wheat I.) ...	0·14	0·10	0·68	1·37	53·54	4·16	2·70
Bread (whole wheat II.) ...	0·05	0·32	0·23	2·36	53·62	4·34	2·42
Bread (wheat flour I.) ...	0·06	0·37	0·27	1·99	64·81	0·00	0·34
Bread (wheat flour II.) ...	0·15	0·38	0·91	1·74	64·12	0·00	0·17
Corn-cake (maize) ...	0·16	0·19	0·00	2·80	61·74	3·54	2·22

C. A. M.

Determination of Caffeine in Coffee. A. Forster and R. Riechelmann. (*Zeit für öffent. Chemie*, iii., 235.)—The authors traverse the statement of Juckenack and Hilger (ANALYST, this volume, p. 190), that the use of soda in their method for the determination of caffeine vitiates the results. Having made four determinations in the same coffee extract by their method, two with and two without the use of soda, they found:

	With Soda.	Without Soda.
1.	5·21 per cent. caffeine.	5·18 per cent. caffeine.
2.	5·24 ,, ,,	5·24 ,, ,,

The use of soda promotes a clean separation of the aqueous solution from the chloroform, without any intermediate emulsion. H. H. B. S.

Estimation of Caffeine in Kola, Guarana, and Coffee. C. H. La Wall. (*Amer. Jour. Pharm.*, 1897, lxix., 350, 351.)—By this, which closely resembles the Keller process for tea, the use of the Soxhlet apparatus is obviated, and the results obtained in a much shorter space of time. Five grammes of the sample are mixed with 5 c.c. of 10 per cent. ammonia solution, and after being left for thirty minutes the alkaloid is shaken out with chloroform in three successive portions of 20 c.c., emulsification being avoided by adding a little powdered magnesium car-

bonate. The mixed chloroform washings are transferred to a weighed flask, the solvent evaporated, and the residue, consisting of fat and alkaloid, weighed. The fat is then extracted with warm ether, the successive fractions being decanted each time without loss of caffeine, and the residue again dried and weighed. In the case of kola, the ether also removes the theobromine; but this being but a small percentage, may be ignored.

The results thus obtained with kola-nuts showed 1·37 to 1·48 per cent. of caffeine as against 1·39 per cent. by extraction with chloroform in a Soxhlet apparatus. With two samples of guarana the corresponding figures were 4·62 to 4·68 and 4·32 per cent. respectively. C. A. M.

Estimation of Alkaloids in White Hellebore. C. H. La Wall. (*Amer. Jour. Pharm.*, 1897, lxix., 351, 352.)—Keller's assay process was found to give satisfactory results. Ten grammes of white hellebore are mixed with 25 grammes of chloroform, 75 grammes of ether, and 10 grammes of 10 per cent. ammonia solution in a flask, which is well shaken and allowed to stand for six hours or overnight. Five c.c. of 10 per cent. ammonia solution are then added, and after being well shaken, 50 grammes of the clear solution (=5 grammes of white hellebore) are poured off into a separating funnel, and the alkaloid extracted with acidulated water, 3 fractions of 20 c.c. each being used. The aqueous washings are transferred to another funnel, made alkaline with ammonia, and the separated alkaloids shaken out with a mixture of chloroform and ether (3 : 1). This extract is placed in a weighed flask, the solvent evaporated, and the residue dried and weighed.

Five different samples of the commercial drug, in the form in which it is sold for an insecticide, when thus assayed, yielded from 1·12 to 1·15 per cent. of alkaloids, while a sample of the whole drug ground gave 1·75 per cent. The author considers that 1 per cent. would be a fair limit for the minimum yield of alkaloids by this process. C. A. M.

The Estimation of Menthol in Oil of Peppermint. L. F. Kebler. (*Amer. Jour. Pharm.*, 1897, lxix., 189-195.)—Power and Kleber were the first to adapt the acetylation test for fixed oils to the examination of oil of peppermint (*Pharm. Rundschau*, xii., 157), but as their method requires about a day, the author has modified it so that the percentage of menthol can be determined in about three hours.

Estimation of Combined Menthol.—From 10 to 12 grammes of the oil (accurately weighed) are boiled for an hour under a reflux condenser with 12 c.c. of normal alcoholic potash, and the excess of alkali titrated back with standard sulphuric acid, with phenolphthalein as indicator. Each c.c. of alkali consumed corresponds to 0·156 gramme of menthol in the form of esters.

Estimation of Total Menthol.—From 12 to 15 grammes of the oil are boiled for an hour under a reflux condenser with an equal weight of acetic anhydride and 2 grammes of anhydrous sodium acetate. When cool, the mixture is washed twice in a separatory funnel with 150 c.c. of water. After the second washing the aqueous layer is removed, and 50 c.c. of water added, together with a few drops of phenolphthalein solution and sufficient 5 per cent. aqueous potash to just give an alkaline

reaction. More water is then added (about 100 c.c.), the whole contents of the funnel are well shaken, and after the layers have separated, the alkaline aqueous solution is withdrawn. The oily layer is again washed with 150 c.c. of water, and when this has been removed as completely as possible, the acetylated oil is transferred to a boiling-flask, the last portions being washed out with alcohol. From 50 to 60 c.c. of normal alcoholic potash are added, the mixture boiled for an hour under a reflux condenser, and the excess of alkali titrated with normal sulphuric acid. The difference between the amount of menthol found and that obtained in the previous determination gives the amount of free menthol.

The following table gives the results obtained with commercial menthol and with a number of specimens of oil of peppermint, all of which the author believed to be genuine, with the exception of that marked "unknown":

Source.	Specific gravity at 15° C.	Per cent. Menthol as Esters.	Per cent. Free Menthol.	Per cent. Total Menthol.	Number of c.c. distilled from 50 c.c. of Oil between										Residue.
					200 *	200-205	205-210	210-215	215-220	220-225	225-230	230-235	235-240	240-250	250-260	
					† 204·8	204·8-209·9	209·9-214	214-219·1	219·1-224·2	224·2-229·3	229·3-234·4	234·4-239·44	239·44-244·56	244·56-254·7	254·7-264·84	
Commercial Menthol	—	none	99·66	99·66	[M.P. 43°]	[B.P. 212°]	—	—	—	—	—	—	—	—	—	
Western ...	0·9112	3·72	29·02	32·48	2	3	4	5	7	3	5	4	2	3	6	6
Michigan	0·9065	3·06	28·25	31·33	2	3	6	9	11	7	4	3	—	—	—	5
,,	0·9147	4·51	29·92	34·43	2	4	5	6	7	4	3	3	3	2	4	7
New York	0·9143	8·07	44·83	52·90	6	9	10	9	7	4	1	1	—	—	—	3
,,	0·9099	7·31	45·43	52·74	11	6	17	11	2	1	—	—	—	—	—	2
Michigan...	0·9099	10·00	40·87	50·87	5	8	15	10	8	2	—	—	—	—	—	2
Unknown	0·8937	8·30	14·94	23·24	30	1	2	3	2	1	1	1	1	—	—	8
Michigan...	0·9279	16·06	31·55	47·61	6	7	7	7	5	4	8	—	—	—	—	6
Mixture of Michigan and New York ...	0·9079	4·68	38·30	42·98	4·5	4·5	9	11·5	10	4·5	2	—	—	—	—	4

* Degrees Centigrade, uncorrected. † Degrees Centigrade, corrected.

C. A. M.

On Commercial Acacia. J. H. Schroeder. (*Amer. Jour. Pharm.*, 1897, lxix., 195-199.)—The United States Pharmacopœia directs that this gum shall be the product of *Acacia Senegal*, and shall comply with the following tests:

(*a*) It shall be soluble in two parts of water, shall give a gelatinous precipitate with basic lead acetate, ferric chloride, or a concentrated solution of sodium borate, and shall not reduce alkaline cupric tartrate.

(*b*) It shall not be colored blue (starch) or red (dextrin) with iodine solution.

The author has examined fifteen representative samples of powdered gum arabic, and although free from dextrin or starch, they all caused a reduction of Fehling's solution at a temperature below 100° C.

Another test (not official) for dextrin is to add to 6 c.c. of a 20 per cent. solution of the gum 3 c.c. of a solution composed of fifteen drops of a concentrated solution of

potassium ferricyanide, five drops of hydrochloric acid (specific gravity, 1·165), and 60 c.c. of water. When dextrin is present a blue colour is said to be produced in about twenty minutes. The author was unable to obtain satisfactory results with this test, as powdered gum arabic free from dextrin showed the blue colour within fifteen minutes. He suggests that changes may occur during the drying of the gum which account for the reactions with Fehling's solution and the ferricyanide solution.

C. A. M.

The Composition of Orris Root. S. A. Tucker. (*Amer. Jour. Pharm.*, 1897, lxix., 199, 200).—Commercial orris root is the rhizome of *Iris Florentina*, deprived of its cortex and dried in the sun. It has a delicate aroma, and when granulated or powdered is used in the manufacture of perfumes, floral extracts, and tooth powders.

In a specimen of the prepared rhizome examined by the author, 1·34 per cent. of fat and wax was extracted with petroleum spirit, whilst 4·13 per cent. of the total substance was soluble in absolute alcohol. The aqueous extract amounted to 14·02 per cent., including 8·31 per cent. of glucose, 1·27 per cent. of sucrose, and a small amount of substances precipitable by alcohol, but no tannin. The odoriferous principles were soluble in alcohol, but not in water. The total organic matter dissolved by water made alkaline with sodium hydrate was 30·30 per cent., consisting for the most part of albuminous and mucilaginous matters, which were precipitated on adding acetic acid and a large volume of water.

Starch was present to the extent of 16·85 per cent.; the moisture was 8·74 per cent., and the ash 2·12 per cent. The latter contained calcium, magnesium, and potassium as carbonates, chlorides, and phosphates. The cellulose and undetermined substances amounted to 10·37 per cent.

C. A. M.

TOXICOLOGICAL ANALYSIS.

Delicacy of Marmé's Test for Alkaloids. S. Verven. (*Ann. Pharm.*, 1897, xiii., 145; through *Chem. Zeit. Rep.*, 1897, 116.)—The tests were carried out by shaking 5 c.c. of a solution of the alkaloid made slightly acid with sulphuric acid, together with 1 c.c. of a solution containing 5 grammes of cadmium iodide and 10 grammes of potassium iodide in 100 c.c. of water. The figures recorded in the first column represent the maximum dilution at which a faint precipitate could be seen; and in the second the point where no precipitate was obtained, even after several minutes:

Atropine	1,600	1,900
Veratrine	5,400	8,200
Aconitine	13,700	27,400
Brucine	14,600	18,200
Cocaine hydrochloride	16,900	20,800
Cinchonine	18,400	24,600
Strychnine	19,200	32,000
Quinine	32,300	37,700

F. H. L.

Detection of Arsenic in Forensic Work. E. Fricke. (*Chem. Zeit.*, 1897, xxi., 303.)—According to the author, the faint volatile "mirror" which occasionally appears in the reduction-tube of the Berzelius-Marsh apparatus, even when the material under examination is free from arsenic, and every care has been taken in the selection of the reagents, is due to the presence of carbon in the zinc employed. The nascent hydrogen may conceivably attack this carbon, producing compounds which are again split up on reaching the hot part of the tube. Fresenius has noted the formation of a similar mirror consisting of silicon; but as this is non-volatile, it cannot be confused with arsenic.

In order to avoid this possible source of error, and also to simplify the process of detecting arsenic generally, Fricke dissolves in ammonium sulphide the precipitate which is always produced by sulphuretted hydrogen in the original liquid after the organic matter has been destroyed, evaporates the solution to dryness, fuses the residue with soda and nitre, and boils the aqueous extract with sulphuric acid. If on being treated again with sulphuretted hydrogen this solution remains clear, arsenic is absent; but if a yellow precipitate is formed, it may then be submitted to Marsh's test for further corroboration. F. H. L.

ORGANIC ANALYSIS.

Examination of Bees-wax. S. Weinwurm. (*Chem. Zeit.*, 1897, xxi., 519).—The author does not consider that Benedikt and Mangold's modification of Hehner's process (wrongly attributed to Hübl) has any advantages over the original form; for it is more complicated, and the difficulty of completely saponifying a wax, even when adulterated with ceresin or paraffin, has been greatly exaggerated. The latter operation can be effected in a covered flask on the water-bath in half an hour, the alcohol being subsequently evaporated off. Benedikt has referred to the "abnormal" acid number 23 and saponification number 90·6, which give a low Hübl ratio. Genuine samples are often seen with an acid number of 24; and this rises to 25 or 26, when they contain a small amount of stearic acid, which is probably not put in as an adulterant, but arises from the use of artificial combs partly composed of the material in question. On the other hand, the acid number may fall to 17·8, with a saponification number of 92·3, and a ratio of 4·2, whence it follows that the determination of these constants is not sufficient to ensure freedom from sophistication. Moreover, the employment of ceresin, stearin, tallow, etc., singly is rapidly diminishing, the substance commonly added being a mixture of stearic acid, Japan-wax, and ceresin, so proportioned as to give the correct figures. It is customary, also, to test for stearic acid, rosin, and glycerin. The first can be detected if it amounts to 1 per cent., rosin only in somewhat larger quantities, while an equal amount of glycerin cannot possibly occur. Determinations of the melting-point and of the iodine number are quite useless; and if Buisine's method be employed it is requisite to estimate the hydrocarbons, as well as the hydrogen evolved.

Two or 3 per cent. of ceresin or paraffin, or 5 per cent. of rosin, may be discovered qualitatively as below. Five grammes of filtered wax are saponified in 25 c.c. of

seminormal alkali and the alcohol removed, 20 c.c. of glycerin are run in, the whole warmed in the water-bath till solution is effected, and 100 c.c. of boiling water added. Pure wax gives a clear, transparent or translucent solution, through which ordinary printed matter can be read with ease. Five per cent. of ceresin or rosin yields a cloudy liquid, and the print is no longer legible; 8 per cent. of ceresin causes a decided precipitate. If the solution is clear, 3 per cent., or, if it is opaque, 2 per cent. of ceresin is added to another sample of the wax and the saponification repeated, when from the appearance of the soap solution the presence or absence of either impurity may safely be deduced. F. H. L.

Proposals with the Object of Establishing Uniformity of Procedure in Determining the Saponification and Acid Numbers. R. Henriques. (*Zeit für öffent. Chemie*, iii., 236.)—As the absence of uniformity in the procedure of different chemists in determining the saponification and acid numbers has frequently led to differences in results, the author puts forward suggestions, founded upon his own experience, with the object of promoting more general agreement.

Saponification Number.—The quantity of substance to be taken from the determination should be 3 to 4 grammes, and not 1 to 2 grammes, as originally proposed. It is immaterial whether potash or soda be used for the saponification, provided the results are expressed in terms of KHO; but the author prefers soda, as sodium carbonate is very nearly insoluble in alcohol of 95 to 96 per cent. For this reason, the alcoholic solution should, by preference, be made by dissolving the sodium hydrate in alcohol direct, instead of by first dissolving in water and then adding the alcohol.

For warm saponification, 3 to 4 grammes should be placed in a flask fitted with a reflux condenser, and either 25 c.c. $\frac{N}{2}$ alcoholic alkali added or 25 c.c. normal alcoholic alkali and 25 c.c. alcohol. The flask should then be boiled briskly for from half an hour to an hour, and the excess of alkali titrated back with $\frac{N}{2}$ hydrochloric acid.

For the cold saponification of oils and fats, 3 to 4 grammes should be dissolved in 25 c.c. petroleum spirit in the cold, and allowed to stand for twenty-four hours at the ordinary temperature. Waxes should be treated thus: Dissolve by warming in 25 c.c. petroleum benzine (boiling at 100° C.); then add the alkali, and again warm for a few moments to ensure complete solution.

Acid Number.—The quantity taken may be the same as in determining the saponification number, though in certain cases, as, for instance, with lubricating oils, it is better to take as large a quantity as possible—say, 5 to 10 grammes. The author dissolves, as a rule, in petroleum spirit or ethylic ether, adds half the volume of alcohol, and titrates with aqueous $\frac{N}{2}$ soda solution.

With regard to the expression of the results, the author proposes that for the acid number, as well as for the saponification number, Koettstorfer's method of calculation should be adhered to. H. H. B. S.

On Solid and Liquid Siccatives. H. Amsel. (*Zeit. angew. Chem.*, 1897, 303-306 and 344-347.)—In this paper the author gives a description of his further work on this subject, and replies to some of Weger's minor criticisms (ANALYST, xxi., 261 and 300). The method of analysis now recommended is a combination of his own with that proposed by Weger, and comprises the determination of mineral matter (free and combined), moisture, solubility in chloroform and alcohol, acid value, saponification value, and ammonia reaction.

The total mineral matter is estimated in the ash, and the insoluble mineral matter in the portion insoluble in chloroform, as described by Weger. The acid value is determined in alcoholic mixture and also in chloroform, the latter giving a higher result, and not the true acid value.

The ammonia reaction consists in shaking the resinate with 10 per cent. ammonia, filtering and adding acid or water, when, if resin be present in excess, there should be a precipitate. But even with 2·5 per cent. ammonia, the author obtained precipitates with samples in which he considered the resin present entirely in the combined state. As pure colophony is soluble in the cold in 1 per cent. ammonia, experiments were made with that strength; but though the results were more satisfactory, they were by no means certain.

The moisture is determined by drying at 90°-95° C. In the author's samples it varied from 0 to 33 per cent., the usual amount being from 2 to 6 per cent.

A direct determination of the resin acids by Laudin's modification of Twitchell's process (*Chem. Zeitung*, 1897, iv., 25) has not, so far, given satisfactory results.

In analysing liquid siccatives, the solvent (turpentine-oil) is determined by drying a weighed quantity at 150° C., the mineral matter in the ash, and the acid value in alcoholic mixture. The acid value will often distinguish between metallic linoleates and resinates. Thus a liquid siccative prepared from manganese linoleate gave 45, whilst one from manganese resinate showed 100. They also behave differently with regard to the end-point of the titration, the red colour rapidly disappearing in the case of metallic resinates. A practical test should also be made by mixing a pure linseed-oil with 5 to 10 per cent. of the siccative, and noting the time required for drying. C. A. M.

On the Behaviour of Shellac Resin Acids in Gladding's and Twitchell's Methods. F. Ulzer and R. Defris. (*Zeit. anal. Chem.*, 1897, xxxvi., 24-27.)—In the separation of fatty and resin acids by Gladding or Twitchell's method the authors found that the resinous acids of shellac behaved quite differently to the ordinary resin acids. For example, a sample of dark shellac which contained 0·05 per cent. of unsaponifiable matter yielded by Gladding's process only 13·76 per cent. of "resin acids," so that in this respect they appeared to be more nearly related to the fatty acids than to the genuine resin acids. The silver salts of the shellac acids differed from the colophony acids by being practically insoluble in ether.

The acids of the brown shellac gave by Twitchell's method 66·56 per cent. of "resin acids" not esterified by the passage of the hydrochloric acid gas through the alcoholic solution. By shaking out the petroleum spirit solution of the acids and esters

with dilute sodium hydrate, an ester was obtained which formed a light-yellow semi-transparent resin-like mass which had a saponification number of 199·5.

The acids of an orange shellac, having acid number 53·05 and saponification number 200·98, gave by Twitchell's process 72·89 per cent. of resin acids which could not be esterified. Therefore the shellac acids appear to behave to some extent like fatty acids, since part of them form esters on being treated with hydrochloric acid gas in acid solution.

Specimens of Angola-Copal and Kauri-Copal when examined by Twitchell's process showed respectively 86·01 and 86·37 per cent. of resin acids.

See also ANALYST, xvi., 169, and xx., 60. C. A. M.

Estimation of Guaiacol by Demethylation. M. Adrian. (*Arch. de Pharm.*, 1897, ccxxxiv., 286.)—The process here described is a modification of that of Béhal and Choay (*Jour. Pharm. Chim.* [5], xxvii., 254). One hundred grammes of the sample to be examined are placed together with 10 c.c. of water in a 250 c.c. flask. This is connected with a flask in which hydrobromic acid is liberated and which has an outlet tube connected with a reflux condenser. A bulb-tube is placed between the flask containing the guaiacol and the generating flask, in order to prevent any liquid being sucked back into the latter during cooling, and the gas, after leaving the reflux condenser, passes through two wash-bottles containing water. The hydrobromic acid is generated by the action of water on phosphorus tribromide, and is passed slowly through the guaiacol, which is gently warmed after thirty minutes. The demethylation is complete in less than an hour with poor samples of guaiacol, but usually an hour and a half is required.

When cool, the contents of the flask are diluted with 500 to 600 volumes of water, and distilled by means of a current of steam to remove volatile substances (monophenols). The pyrocatechin and homopyrocatechin in the residue are extracted with ether, the solvent slowly evaporated, and the residue dried. The two substances are separated by means of benzene, in which the former is completely soluble, and can be crystallized out and weighed. From the amount of pyrocatechin the quantity of guaiacol in the sample can be calculated within 5 or 6 per cent. It is essential that the benzene should contain not the slightest trace of moisture or the crystallization of the pyrocatechin will be incomplete; and even with perfectly dry benzene the mother liquor must be concentrated and a second crystallization made. C. A. M.

The Detection of α-Naphthol in the Presence of β-Naphthol. E. Leger. (*Jour. Pharm. Chim.*, 1897, v., 527-529.)—The reagent is a freshly-prepared solution of sodium hypobromite, made by mixing 30 c.c. of soda solution (36° Bé) with 100 c.c. of water, and then adding 5 c.c. of bromine, and shaking.

α-Naphthol in a saturated aqueous solution gives with the above reagent a dirty-violet coloration, or even precipitate, which is still pronounced when the saturated solution is diluted with nine volumes of water.

β-Naphthol in saturated solution gives a yellow colour, passing to green and

then back again to yellow. If the saturated solution be diluted with its own volume of water, and two drops of the hypobromite solution added, a yellow colour is produced, but this, on shaking the liquid, immediately disappears.

In testing a mixture, a saturated solution is to be prepared, diluted with its own volume of water and 2 drops of the reagent added. The presence of 1 part of α-naphthol in 100 parts of β-naphthol is thus easily recognised. C. A. M.

The Methods of Testing Rose-Oil. R. Jedermann. (*Zeit. anal. Chem.*, 1897, xxxvi., 96-102.)—From an extended experience of the properties of genuine rose-oil, the author considers that the solidifying temperature and the sulphuric acid test are not of much value. The latter consists in mixing 5 drops of the oil with 25 drops of concentrated sulphuric acid, and then adding 10 c.c. of alcohol (specific gravity 0·8). With geranium-oil the solution at once becomes turbid, whilst with genuine rose-oil it is said to remain practically clear and colorless. Few genuine specimens of East Roumelian oil, however, remain clear, as traces of resinous matter distilling over with the oil often cause turbidity.

The solidifying point, which in genuine oils may deviate in either direction from the limits 17·5° to 20° C., is equally inconclusive, and the author places more reliance on the refractive index. The average for rose-oil at 30° C. is 43° 30′, that of geranium-oil being 41° 30′. The specific gravity may also give some indication as to the purity of an oil. At 30° C. this constant in genuine rose-oil varies between 0·8555 and 0·8645, and in geranium-oil between 0·883 and 0·8895. But in the author's opinion the most reliable tests are the expert judgment of the odour and the practical results obtained in the use of the oil (*cf.* ANALYST, xx., 48). C. A. M.

Estimation of Carbon Disulphide in Alcohol, Carbon Tetrachloride, etc., and a Method for the Purification of the latter. W. Schmitz-Dumont. (*Chem. Zeit.*, 1897, xxi., 487 and 510.)—Although the processes for the estimation of carbon disulphide in alcohol based on its conversion into potassium xanthate can be carried out smoothly, especially if the product is oxidized with solid permanganate and the sulphuric acid determined by means of barium chloride, yet they are not trustworthy because the initial reaction between the disulphide and the excess of caustic potash is not quantitative. If 10 c.c. of impure alcohol, containing about 0·1 gramme of CS_2, are warmed to 50° or 60° C. for twenty minutes with 20 c.c. of alcoholic lead acetate and 10 c.c. of normal alcoholic potash, the whole of the sulphur is recovered as lead sulphide, which may be oxidized to sulphate by bromine and nitric acid, dissolved in sodium acetate, and precipitated as barium sulphate. The results are quite satisfactory, and the scheme would probably work equally well with carbon tetrachloride; but the author finds on the whole that the method described below is quicker and better.

Processes depending on the use of phenylhydrazine are objectionable (1) in the case of benzene, etc., because of the relatively large solubility of the precipitate in the alcohol employed, which causes the figures obtained to be too low; and (2) in the case of carbon tetrachloride they are useless owing to the simultaneous formation

of a crystalline normal or basic hydrochloride of the reagent—both of which bodies are described fully in the original paper.

The following process is available for the estimation of CS_2 in all liquids or gases which can be brought into contact with alcoholic silver solution and aniline, and which do not contain ingredients liable to prevent the formation of AgS. To every 10 c.c. of the suspected fluid are added 50 c.c. of a 5 per cent. alcoholic solution of silver nitrate and 5 c.c. of aniline, and the mixture is heated for half an hour at 50° or 60° C. When the amount of CS_2 present is absolutely unknown, a considerable excess of aniline may be introduced (1 gramme $CS_2 = 2{\cdot}44$ grammes, or 2·51 c.c.; 1 c.c. $CS_2 = 6{\cdot}41$ grammes, or 6·6 c.c. of $C_6H_5NH_2$), and the silver added in instalments so long as a precipitate forms, the liquid being warmed each time until it becomes clear. There is little fear of loss by proceeding in this manner, but, if desired, the operation can be repeated when the necessary proportions have been arrived at. The silver sulphide, contaminated with metal reduced by the aniline, is thrown on a very small asbestos filter, washed with alcohol and ether by the aid of the pump, and melted with sodium carbonate and nitre. The melt is dissolved in water, acidified with nitric acid, diluted considerably, and the S determined with barium. The yield approaches closely to the theoretical, but great care must be taken regarding the purity of the different reagents. Gases should be passed through the mixed silver solution and aniline maintained at a temperature of about 60°, the absorption being complete.

In order to remove CS_2 from commercial CCl_4, $1\frac{1}{2}$ times as much caustic potash as is needed to combine with the disulphide is dissolved in an equal weight of water, and, together with 100 c.c. of alcohol, introduced into 1 litre of the chloride. After heating to 60° for thirty minutes, the xanthate is extracted by the addition of water, the aqueous liquid run off, and the entire process repeated twice again with half the original amount of alkali. Finally, 500 c.c. of water are shaken with the chloride until the separation is rapid and complete, the product dried with solid caustic potash, or preferably, metallic sodium, and distilled over paraffin to eliminate the C_2Cl_6. So prepared, it gives no sulphur precipitate with silver and aniline, and it boils constantly at 76·3° C. at 760 mm., the thermometer being in the vapour.

F. H. L.

Estimation of Nitrogen in Guanos. V. Schenke. (*Chem. Zeit.*, 1897, xxi., 490.)—The experiments on which the author's last communication on this subject (ANALYST, xxii., 77) was based having been conducted on old samples of guano, he has repeated them on fresh specimens coming from Peru and Damaraland. His conclusions are the same as before, especially as concerns the Haselhoff extraction process, which, although it can be modified so as to give useful results, thus becomes too tedious for practical work.

Schenke now calculates the nitrate nitrogen by deducting the quantity yielded by the Kjeldahl process from the total; for some of the nitrogen in uric acid is converted into ammonia on distillation even with magnesia, and therefore the difference between this amount of soluble nitrogen and that obtained by employing caustic soda.

or potash, as in Ulsh's reduction method, cannot truly represent the nitrogen present as nitric acid. F. H. L.

A Substitute for Hide Powder for Use in the Determination of Tanning Material. W. Schmitz-Dumont. (*Zeit. für öffent. Chemie*, iii., 209.)—The author refers to the difficulty of preparing hide powder of good uniform quality, which is so essential to its successful use in estimating the value of tanning agents, and describes experiments made with the object of finding a substance more suited to the purpose.

Experiments made with gelatin hardened with alum and chromic acid not proving successful, the author tried formalin-gelatin, prepared as follows: Thick filter-paper was saturated with a 10 per cent. gelatin solution, and laid upon glass rods to dry. When dry, it was immersed for twenty-four hours in a 2 per cent. formalin solution, and afterwards dried at 95° C. It was then cut up into strips, which were further reduced to powder by being passed through a mill. It was then again immersed for twenty-four hours in a 2 per cent. formalin solution, the fluid pressed out, and the powder dried at 100° C. until 50 grammes, dried separately, lost only about 0·05 gramme. The preparation was now freed from trioxymethylin by digestion in hot water in a Koch's extraction apparatus, until 100 c.c. of the effluent gave no formaldehyde reaction with alkaline resorcin solution. The water was then pressed out and the powder dried in a water-bath. The finished preparation, which yielded quite an unimportant quantity of soluble matter on digestion in water, was compared with a good hide powder by means of parallel experiments, and, so far as these were taken, with very favourable results. H. H. B. S.

Analysis and Detection of Artificial Silk. P. Truchot. (*Ann. de Chimie Analyt.*, ii., p. 121.)—Artificial silk, like natural silk, when treated with Schweitzer's reagent, swells up and dissolves, but is distinguished from the latter by a precipitate of cellulose forming on dilution, or on the addition of hydrochloric acid.

The deep yellow, syrupy solution obtained by the action of 60° B. (S.G. 1·712) sulphuric acid turns dark-blue in presence of diphenylamine sulphate. This reaction, as well as the rose coloration produced by brucine sulphate, indicates the presence of nitric acid due to the incomplete reduction of trinitrocellulose. It is an extremely delicate test, and can also be performed directly on the fabric; all the artificial silk threads of which, when dipped in diphenylamine sulphate solution, turn to a deep blue, leaving those of the natural silk unaffected.

The specific gravity of artificial silk is 1·490, that of the natural undyed fibre being 1·367 to 1·357. The sample examined by the author contained 1·52 per cent. of ash and 0·23 per cent. of nitric acid, corresponding to 1·63 of tri-nitrocellulose. C. S.

Estimation of Ash in Crude Sugars. Schrefeld. (*Zeits. Rubenz.*, xlvii., 560; through *Chem. Zeit. Rep.*, 1897, 149.)—The author finds that the ash of sugar loses weight in a muffle at a temperature of 700° C., and that at 850° or 900° it melts and begins to volatilise. Some of the constituents of the ash are, therefore, more volatile than the potassium sulphate which it contains. F. H. L.

INORGANIC ANALYSIS.

A Reaction of Carbon Monoxide. A. Mermet. (*Rev. de Chimie Analyt.*, v., pp. 130-133.)—The author finds that a weak solution of potassium permanganate, acidified with nitric acid, is decolorized by carbon monoxide, the reaction being more rapid in presence of silver nitrate. The time varies between one and twenty-four hours for air containing 0·2 to 0·02 per cent. of carbon monoxide.

The silver nitrate solution should contain 2 to 3 grammes of the crystallized salt per litre. The permanganate solution is prepared by boiling 1 litre of distilled water with a few drops of pure nitric acid, and destroying any organic matter present by adding a few drops of permanganate solution. When cold, 1 gramme of permanganate crystals and 50 c.c. of pure nitric acid are added, and the solution stored away from light and dust.

The reagent is prepared, immediately before use, by mixing 20 c.c. of the silver nitrate solution with 1 c.c. of the permanganate solution, 1 c.c. of pure nitric acid, and 28 c.c. of distilled water freed from organic matter. To apply the test, the reagent is poured into a flask containing the air under examination; or—since dust or reducing gases may also decolorize the reagent—the air is purified by filtration and passage through absorptive reagents, so that, the presence of a reducing agent being detected by the first method, its nature may be ascertained by the second. The presence of sulphur is detected by the brown tinge due to silver sulphide imparted to the reagent.

In testing suspected air—*e.g.*, from a mine—the sample is collected in the usual manner, the inlet of the bottle being provided with a cotton filter in the case of a dusty atmosphere. A check experiment is performed with normal air. C. S.

Estimation of Oxygen in Illuminating Gas. O. Pfeiffer. (*J. Gasbeleucht*, 1897, xl., 354; through *Chem. Zeit. Rep.*, 1897, 148.)—The amount of oxygen in 100 c.c. of coal-gas can be determined colorimetrically in a Bunte burette over 5 c.c. of caustic potash by adding 0·2 gramme of pyrogallol dissolved in 2 or 3 c.c. of oxygen-free water. The absorption is complete in five minutes, and the colour first darkens and then becomes paler again. It is not altered by ordinary variations in temperature, but changes in the proportions of the reagents affect it. Sulphuretted hydrogen gives it a wine-red shade. If 1 per cent. of oxygen exists in the gas, the colour is produced instantly; but if only 0·25 per cent. is present, the burette requires shaking for five minutes.

To prepare a series of standard tints, 30 grammes of sugar and 30 c.c. of 1 : 3 sulphuric acid are boiled in 1 litre of water for fifteen minutes, and 85 c.c. of 1·3 caustic soda added. This liquid is mixed, drop by drop, with water in test-tubes until the colours match those yielded by artificially-produced mixtures of coal-gas and oxygen treated as above. The standards are permanent if preserved in the dark.

F. H. L.

Simple and Rapid Analysis of Nickel Castings; Separation of Nickel and Cobalt by the von Knorre Method. J. Albahary. (*Rev. de Chimie Analyt.*,

v., 102-104.)—Five grammes of the substance are dissolved in 50 c.c. of nitric acid, evaporated to dryness, taken up by 10 c.c. of dilute sulphuric acid, and boiled for forty-five minutes.

Silica is determined by filtering, igniting, and weighing; *copper* by precipitation on the water-bath by means of 50 c.c. of saturated hydrogen sulphide.

Iron.—After heating to drive off the hydrogen sulphide, the solution is made up to 250 c.c., the iron being thrown down from 100 c.c. by ammonia, and freed from nickel by reprecipitation.

Nickel and Cobalt.—The other 150 c.c. of solution are, after the addition of 5 grammes of sodium sulphate, electrolyzed for five hours by a current of half an ampère, to throw down nickel and cobalt.

Magnesium is estimated in the residual liquid, as ammonio-phosphate.

Separation of Nickel and Cobalt.—The metals deposited on the negative pole are dissolved in dilute sulphuric acid, and the warm solution treated with an excess of a hot saturated solution of nitro β-naphthol in 50 per cent. acetic acid in presence of a little hydrochloric acid to prevent the simultaneous precipitation of nickel. After leaving the precipitate to subside for three hours, and making certain that all the cobalt is thrown down, it is first washed with cold, then with hot, 12 per cent. hydrochloric acid, and finally with boiling water; then dried and ignited with 2 grammes of crystallized oxalic acid in a current of hydrogen, the metallic cobalt being weighed in the crucible. Nickel is estimated by difference or by precipitation with potash, after removing by evaporation most of the acetic acid from the solution.

Sulphur is estimated by the Carius or Campredon method; *carbon* by the Corleiss (chromic acid and copper sulphate) or Viborg method. C. S.

Determination of Phosphoric Acid as Phospho-Molybdic Anhydride. Woy. (*Chem. Zeit.*, xxi., 441.)—By following up the work of Meineke and Hundeshagen, the author has succeeded in perfecting a method by which phosphoric acid in plant-ashes, fertilizers, and raw phosphates can be determined by means of molybdenum, without the usual subsequent precipitation with magnesia-mixture. The method is as follows:

Reagents.—1. A 3 per cent. solution of ammonium molybdate, made by dissolving 120 grammes in 4 litres of water.

2. Ammonium nitrate solution; 340 grammes in 1 litre.

3. Nitric acid of 1·153 specific gravity, containing 25 per cent. HNO_3.

4. Solution for washing; 200 grammes ammonium nitrate and 160 c.c. nitric acid in 4 litres.

Procedure. — 0·1 gramme P_2O_5 requires for the formation of the anhydride $24MoO_3$, P_2O_5, 2·9856 grammes ammonium molybdate, which is equivalent to 100 c.c. of the 3 per cent. solution. Therefore, in working with 0·5 gramme of substance, 5 c.c. of the solution are required for every 1 per cent. P_2O_5; but an excess of 20 c.c. above the theoretical quantity should be allowed. Usually 10 c.c. of nitric acid suffice, but the quantity can be increased to 20 c.c. in certain cases.

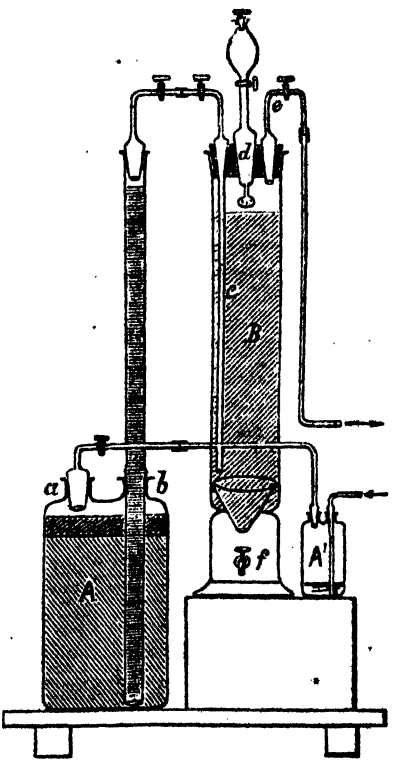
Thirty c.c. ammonium nitrate solution and 10 c.c. or 20 c.c., as the case may require, of nitric acid, are measured together and added to the solution containing

the phosphoric acid. The whole is then heated over a lamp until bubbles commence to rise. The necessary quantity of molybdate solution, also heated to the same extent, is then allowed to flow into the middle of the hot solution, which is meanwhile kept in motion by giving the beaker a circular movement with the hand. The precipitate forms almost immediately, and in ten or fifteen minutes subsides sufficiently to filter off. The author filters through a porcelain Gooch crucible with the assistance of atmospheric suction. The precipitate is washed by decantation with 50 c.c. of hot washing solution, and then redissolved into the same beaker with 10 c.c. of an 8 per cent. ammonia solution. Twenty c.c. ammonium nitrate, 30 c.c. water, and 1 c.c. molybdate solution are then added. The whole is now heated over a lamp as before, and re-precipitated by adding 20 c.c. of hot nitric acid drop by drop, the fluid meanwhile being kept in motion as before. The precipitate soon subsides, and is then filtered off through the same crucible and washed with the washing solution. The incineration is accomplished by placing the porcelain Gooch crucible inside a nickel crucible, and heating gradually until the bottom is of a dull red heat. In about fifteen minutes, at this temperature, the yellow precipitate is completely converted into the black anhydride $24MoO_3$, P_2O_5, containing 3·946 per cent. P_2O_5.

H. H. B. S.

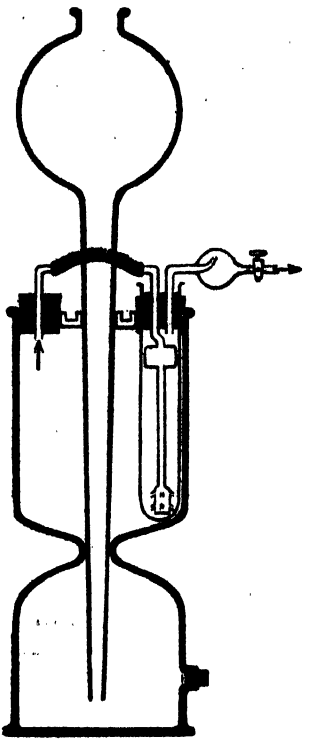
APPARATUS.

A New Apparatus for Purifying Gases. **A. J. J. Vandevelde.** (*Chem. Zeit.*, xxi., 445.)—The apparatus shown in the accompanying illustration allows of gases being easily and freely passed through it, and yet acts as a thoroughly efficient absorber. It also admits of the sulphuric acid used for drying being conveniently renewed. It consists of a large Woulff's bottle A, in which the carbonic acid is absorbed, and a cylindrical vessel B, in which the gases are dried. The Woulff's bottle is three parts filled with solid caustic potash, upon which is a layer of quicklime. The tube *b* contains soda-lime and the small Woulff's bottle A' sulphuric acid. The funnel tube *d* is provided with a stopcock, and at its lower end is a hollow perforated globe. The cylinder B is filled with broken glass moistened with sulphuric acid. The direction in which the gases are passed through the apparatus is shown by the arrows. When the sulphuric acid requires to be renewed, it is drawn off by means of the tap *f*, and a fresh supply introduced through the funnel tube *d*.



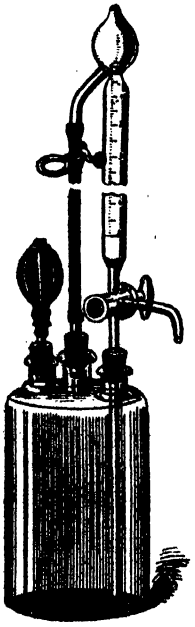
H. H. B. S.

New Gas Apparatus. H. Witzel. (*Chem. Zeit.*, 1897, xxi., 536.)—This apparatus combines the functions of the Kipp generator and the ordinary washing or drying-tubes, so that it becomes more convenient to handle. It will also be observed that the purifying vessel is specially designed to prevent any splashing of the liquid employed. F. H. L.



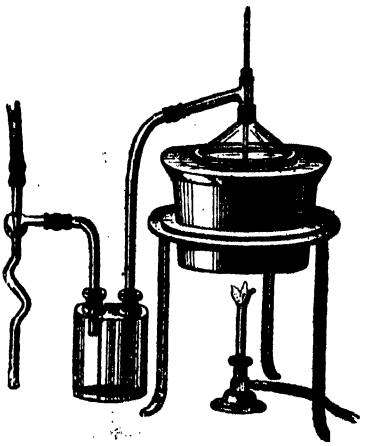
WITZEL'S GAS APPARATUS.

New Laboratory Apparatus. (*Chem. Zeit.*, 1897, xxi., 351.)—*Peters and Rost's Automatic Burette.*—This apparatus can either be made to carry a burette or a pipette, but the latter arrangement is more generally useful. To fill the measuring vessel, the pinch-cock is closed, the tap set so that the burette and vessel are in connection, and the liquid pumped up by squeezing the rubber ball; the tap is then closed and the pinch-cock opened, when any excess of the solution that has accumulated in the top bulb runs back into the bottle, and the 0-point is automatically set. The liquid can then be run off by turning the 3-way tap in the proper direction. F. H. L



NEW LABORATORY APPARATUS.

Haussmann's Vacuum Apparatus. (*Chem. Zeit.*, 1897, xxi., 351.)—This consists of a cylindrical porcelain still provided with a ground lip on which an inverted glass funnel rests, making a joint in the manner illustrated. The apparatus is therefore cheap, simple, and can be used on any water-bath. F. H. L.



HAUSSMANN'S VACUUM APPARATUS.

THE ANALYST.

OCTOBER, 1897.

PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS.

NOTE ON COMMERCIAL GROUND CASSIA.

By W. F. Keating Stock, F.I.C.

(*Read at the Meeting, June* 2, 1897.)

I have analysed a large number of samples of commercial ground cassia during the last three years, and among them I have not found one which corresponded in composition with samples of genuine cassia lignea ground in the laboratory. In the majority of cases the difference was due to the presence of sand and earthy matters generally, these predominating over the natural ash to the extent of from 1 per cent. up to 9 per cent. In two cases exhausted ginger was present, and in several others the sample was entirely composed of *Cassia vera*, which differs considerably in quality and composition from the ordinary *Cassia lignea*.

With regard to the presence of extraneous earthy matter in ground cassia, one is inevitably driven to the conclusion that it does not come there by honest means. For if the nature of the process by which cassia is collected and prepared for market in its unground condition is considered, it is seen to be a cleansing process, and that the presence of sand and dirt is avoidable. This view is supported, I think, by the following analyses made in my laboratory:

Average total ash in genuine *Cassia lignea*	... ...	= 1·92 per cent.
,, ,, in high-class commercial samples	...	= 2·68 ,,
,, ,, in ordinary commercial samples	...	= 5·09 ,,
Variations of total ash found in ordinary samples		= 3·18 – 11·60 ,,

One would not like to say that small variations in the proportion of natural ash are impossible, but the variations cannot by any reasonable argument be made to embrace sand or earthy matter, and when these occur to the extent of more than a few tenths per cent., the sample should be looked upon with suspicion.

The detection of exhausted ground ginger in admixture with ground *Cassia lignea* offers no difficulty to the analyst, since the starch granules contained in each are so entirely dissimilar. But a rapid and accurate method of determining the relative proportions of the two is not, perhaps, so easy to arrive at. The ether extract, alcohol extract, water extract, total ash, soluble ash, and phosphoric acid, all failed

to give me any useful factor. I, however, found a valuable aid to the determination in the proportion of woody fibre in the water-free substance.

The following table will show how the woody fibre supplies the necessary data for calculation:

Samples Analysed.	Moisture.	Woody Fibre in Undried Sample.	Woody Fibre in Water-free Sample.
Commercial ground cassia (five samples) ...	12·17	24·46	27·70
Laboratory ground cassia	16·85	22·56	27·10
Suspected sample	13·23	15·33	17·60
Exhausted ginger	11·84	5·03	5·70

If C represents the cassia in a dried sample, D the woody fibre in the sample, and E the fibre in dried exhausted ginger, then C is obtained as follows:

$$C = \frac{(D - E)100}{27{\cdot}5 - E}.$$

The proportion of cassia so obtained is *dry* cassia, and must be corrected for the moisture present in the sample.

Owing to the importance of the fibre in the foregoing formula, it must, of course, be determined with great care, and, without going into much detail, I may mention that in my practice 3 grammes of finely-ground cassia are boiled in a 600 c.c. flask attached to a reflux condenser, with 200 c.c. of dilute sulphuric acid, containing 1·25 per cent. of ordinary strong acid, for thirty minutes. The liquid is then filtered through a linen-cloth filter, using a filter-pump; the residue is washed free from acid with boiling water, returned to the flask, boiled again for thirty minutes with 200 c.c. of a 1·25 per cent. solution of sodium hydrate. The flask is then filled up with cold water, and allowed to stand for twenty minutes, when the liquid is again filtered through the same cloth filter, and the fibre is thoroughly washed with boiling water. Finally it is treated with 50 c.c. of dilute hydrochloric acid, containing 12·5 c.c. of the ordinary pure acid. It is washed free from every trace of acid with boiling distilled water, transferred to weighed watch-glasses, dried to constant weight, ignited, and any mineral matter deducted.

The substitution of *Cassia vera* for *Cassia lignea* is a matter which cannot in the existing state of things be dealt with under the Sale of Food and Drugs Acts. It is generally admitted—and, indeed, it is quite obvious—that both in flavour and convenience in use *Cassia vera* is inferior to *Cassia lignea;* but since it is "cassia," no penalty attaches to its substitution for *Cassia lignea* unless *Cassia lignea* has been specifically demanded.

I have drawn attention to this variety of cassia in the interests of Public Analysts who may not yet have met with it, and to whom it might prove misleading. It differs from *Cassia lignea* in two important points. In the first place, it contains much larger starch granules, and, in the second place, it yields with cold water a tenacious glairy mass, owing to its content of mucilage. The starch granules of *Cassia vera* do not differ greatly in their larger forms from those of exhausted ginger,

and it behoves the Analyst meeting with *Cassia vera* in admixture with *Cassia lignea* not to depend too much upon the microscope, but to look closely into the general characters of the sample.

Some analyses of commercial samples of *Cassia vera* have been made in my laboratory, and the figures so obtained are contrasted with similar figures for *Cassia lignea* in the following table:

Constituent Sought.	*Cassia lignea* (several samples).	*Cassia vera* (several samples).
Moisture	12·17	11·15
Total ash	5·36	5·15
Soluble ash	1·20	1·45
Phosphoric acid	·19	·13
Woody fibre in water-free sample	27·70	19·50

NOTE ON THE BACTERIOLYSIS OF GELATIN.

By Dr. S. Rideal, D.Sc., and R. Orchard.

(*Read at the Meeting, June* 2, 1897.)

In ordinary bacteriological work for water analysis and other purposes, free use is made of gelatin media, and one of the distinctions between bacteria is founded on their liquefying, or not liquefying, gelatin. The jelly runs down the test-tube or collects on the surface of the plate, and forms a pool, which is fluorescent, or colored, or possessed of odours, according to the nature of the pure culture present.

Although an amount of work has been published on the changes resulting from the decomposition of various organic matters, especially albumin, no record of any experiments, or series of experiments, could be found which demonstrated quantitatively the chemical changes that take place when the ordinary gelatin medium used in bacteriological investigations is liquefied; in fact, the published analyses of commercial gelatin itself were very imperfect.

With the object, therefore, of determining the changes produced when gelatin is in process of liquefaction by bacteria, we have made a series of experiments on the following lines:

A quantity of gelatin medium was prepared, containing 10 per cent. of "Coignet's best gelatin" and 10 per cent. of Pasteur's solution, made up with Rochelle salts in the place of ammonium tartrate, in order to avoid the introduction of ready-formed ammonia; this was made faintly alkaline with sodium carbonate, and clarified in the usual manner. After heating the solution to 30° C., so as to be quite fluid, quantities of 20 c.c. were pipetted into 6 oz. conical flasks, plugged with cotton-wool, and subsequently sterilized by heating to 100° C. on three successive days. These were then inoculated from an active culture of the organism, and incubated at "room temperature" for the given time. Conical flasks were employed, in order that the liquefaction of the gelatin should proceed at the maximum rate,

and consequently the organisms were cultivated as far as possible under aerobic conditions. Petri's dishes were tried, but, owing to the difficulty of manipulation without loss when the medium was fluid, they were abandoned. The different flasks showed progressive liquefaction of the gelatin according to time. The liquid produced was in all cases alkaline, had a slight putrefactive odour, was turbid, and contained no sulphuretted hydrogen.

In the subsequent analyses, after the different periods, the contents of the flasks were warmed to the same temperature—30° C.—rendered faintly acid with acetic acid, and heated to 100° C., in order to ascertain whether the bacteria in their growth had produced any weighable quantity of cellulose or coagulable albumin. No precipitate was obtained which could be estimated, except in one case—the sixteen days' culture of *B. fluorescens liquefaciens*, when the precipitate by heat amounted to 0·06 per cent. (= 0·01 N). The contents of the flasks were cooled again to 30° C., diluted with sterile water to 250 c.c., and aliquot portions carefully measured out at this temperature for each determination. In this way strictly comparative results were obtained, independent of the slight evaporation from the gelatin medium. The results were controlled in each case by a determination of the total nitrogen.

The gelatin and albumoses were estimated by Kühne and Chittenden's method of precipitation with saturated ammonium sulphate solution, dissolving the precipitate in warm water, dividing, and determining the total nitrogen in one portion, and in the other, very exactly, the SO_3, then subtracting the nitrogen due to ammonium sulphate, a process which we prefer to that of direct weighing of the precipitate.

The albumoses were determined separately by precipitating a portion of the original solution with Stutzer's reagent (an emulsion of copper hydroxide in glycerin and water), washing, and estimating the nitrogen in the precipitate.

The volatile bases were estimated in a portion of the original, by distillation with caustic soda and titration of the distillate. A further portion of the original was precipitated with alcohol, filtered, and the nitrogen content of the filtrate returned as bases and extractives.

The two liquefying organisms selected were: (i.) *B. prodigiosus*, from an active gelatin culture, and (ii.) *B. fluorescens liquefaciens*, freshly obtained from tap-water. P. F. Frankland describes a gelatin-tube culture of *B. prodigiosus* in the following words: "Grows very rapidly, liquefying the gelatin in the form of a conical sack, which soon extends across the top of the tube, and, gradually passing downwards, involves the whole tube. The liquid is very turbid, with an abundant flocculent deposit of intense crimson colour." And a stab culture of *B. fluorescens liquefaciens* (Flügge) as under: "In the path of the needle there is a whitish growth, whilst a funnel-shaped depression, containing liquid gelatin, forms on the surface; the liquefaction gradually stretches across the tube, and finally the whole contents are fluid and of a fluorescent green colour, whilst a white deposit collects." In the initial plate cultivations, after fourteen days at 20° to 21° C., the gelatin was found to be only partially liquefied by the *B. prodigiosus*, so that our attention was chiefly confined to *B. fluorescens liquefaciens*. A number of flasks were inoculated with this organism, the contents being analysed after *one day*, when only a superficial liquefaction of the gelatin had taken place; *two days*, when about half had been liquefied;

three and a half days, when the liquefaction of the gelatin was quite complete; and finally after *sixteen days*.

The following is a tabulated summary of the results obtained:

Calculated as Grammes of Nitrogen per 100 *c.c.*

Incubated at 20° to 21° C.	Total Nitrogen.	Gelatin and Albumoses.	Albumoses.	Ammonia and Volatile Bases.	Bases and Extractives.	Peptone N unaccounted for.	Gelatin.	Albumin and Cellulose.
Blank (i.)	1·242	1·109	0·514	0·003	0·061	0·069	0·595	
,, (ii.)	1·246	—	0·528	0·004	—	—	0·581	
B. prodigiosus, 14 days ...	1·232	1·024	0·409	0·049	—	—	0·615	
B. fluor. liquefaciens, 1 day	1·33	1·141	0·402	0·028	—	—	0·739	
,, ,, 2 days	1·256	0·682	0·297	0·045	0·105	0·424	0·385	
,, ,, 3½ ,,	1·267	0·242	0·135	0·063	0·486	0·476	0·107	
,, ,, 16 ,,	1·26	0·216	0·081	0·168	0·742	0·124	0·135	0·01

It will be noticed from the analysis of the blank that the original dry gelatin contains roughly 50 per cent. of matter precipitable by Stutzer's reagent, so-called albumoses. In the early stages these albumoses seem to be first attacked by the organism; but subsequently their decomposition proceeds at an equal rate with that of the true gelatin.

A notable feature in this research is the small production of ammonia and volatile bases, in view of the fact that substances such as trimethylamine, indol, and skatol have been generally stated in analyses by other observers to be constant accompaniments of the liquefaction of proteids. That this is not accounted for by the volatilization of such bodies during the incubations is proved by the comparative absence of odour and of strong alkaline reaction in the air of the flasks, and in such loss not being indicated by the total nitrogens. Previous investigations have, however, been conducted with "nutrient gelatin," containing other proteids. The quantity of ammonia produced is insignificant, amounting, even after sixteen days' incubation with *B. fluorescens liquefaciens*, to only 0·168 gramme of nitrogen per 100 c.c., corresponding to 0·204 gramme of ammonia.

The figures for the nitrogen unaccounted for, which would be almost entirely peptones, are very interesting. The original gelatin solution containing 0·069 gramme per 100 c.c., this gradually increases as the liquefaction proceeds, until, with *B. fluorescens liquefaciens*, it reaches the maximum figure of 0·476, when the medium is just entirely liquefied, and then diminishes, after sixteen days, to 0·124, showing that the bacillus, after bringing about the decomposition of nearly all the gelatin and albumoses, subsequently attacks the peptone, which has meanwhile been produced, with a corresponding increase of bases and extractives soluble in alcohol.

It is known that certain bacteria produce peptonizing enzymes, and in the case of the *B. fluorescens liquefaciens* the organism appears, as shown above, to subsequently feed on the peptone produced by its enzyme.

In order to ascertain whether the enzyme is capable of liquefying the gelatin

without the aid of bacteria, a large quantity of gelatin medium was inoculated with *B. fluorescens liquefaciens*, and incubated at 20° C. After about five days, when the culture was entirely fluid, some of it was filtered, by a vacuum, through a Pasteur candle into a filtering-flask containing 100 c.c. of 10 per cent. gelatin that was quite sterile. When about 10 to 15 c.c. of the culture had passed through, the filter was removed, the neck of the flask plugged with cotton-wool, and the gelatin warmed to 30° C, so as to be able to mix the filtrate uniformly with the gelatin solution; it was then allowed to set quite firmly, and incubated at 20° C. As after a few hours the gelatin was not quite solid, it was removed from the incubator and kept in a room at about 10° C. In twenty-four hours the contents of the flask were semi-liquid, and after thirty-six hours quite liquid. It was proved to be sterile by microscopical examination and by attempts at subculture. The liquid did not smell putrid, and was not fluorescent; except that it was liquid, it appeared the same as the original gelatin solution. The contents of the flask were then analysed with the following results, calculated into grammes of nitrogen per 100 c.c.:

Total Nitrogen.	Gelatin.	Albumoses.	Ammonia.	Bases and Extractives.	Peptone.	Vol. Org. Acids.
1·232	·359	0·21	Nil	0·469	0·174	Nil.

The liquid was tested for the presence of peptone by dialysing a portion, and adding caustic soda and copper sulphate to the dialysate, when a strong biuret reaction was obtained; with the original gelatin solution similarly treated there was no reaction. That an enzyme is produced by *B. fluorescens liquefaciens* was further confirmed by repeating the above experiment on 15 per cent. gelatin containing Pasteur's solution as before, but not alkalized, when it was found to be liquefied in twenty hours at 17° to 18° C.

AN IMPROVED METHOD OF DETERMINING PROTEID AND GELATINOID SUBSTANCES.

By A. H. Allen and A. B. Searle.

In a paper recently read before the Society of Public Analysts (Analyst, this volume, p. 228), by Dr. S. Rideal and Mr. C. G. Stewart, a process of determining gelatin was described which was based on the treatment of the solution by chlorine, and drying and weighing the resultant precipitate. In the subsequent discussion, one of us suggested that several advantages might be gained by the substitution of bromine for chlorine, the nitrogen in the precipitate being subsequently determined by Kjeldahl's process instead of the precipitate itself being weighed.

We have since made a number of experiments on these lines, and have devised a modified process which materially lessens the time required for the determination of the proteid or gelatinoid matter in a substance, without impairing the accuracy of the result. The following mode of procedure is that which we have found most satisfactory:

A quantity of the solution, containing about 1 gramme of the albuminoid matter, is brought to a volume of about 100 c.c., and treated in a conical beaker with suffi-

cient dilute hydrochloric acid to render the liquid distinctly acid to litmus. Bromine-water is then added in considerable excess, and the liquid stirred vigorously for some time. The yellowish precipitate which separates is flocculent when first formed, but becomes more viscous after stirring, and, in great part, adheres to the sides of the beaker, which, with its contents, is allowed to stand for about half an hour, or until all the precipitate has settled. The liquid is then decanted through an asbestos filter.* The precipitate adhering to the beaker is washed several times with cold distilled water, the washings being poured through the filter. Occasionally, when most of the free bromine has been washed out of the precipitate, the liquid does not filter clear. It is therefore advisable to keep the washings separate from the filtrate, and, if necessary, to wash with sodium sulphate solution or with bromine-water. We make no attempt to weigh the precipitate, preferring to determine the contained nitrogen by the Gunning-Kjeldahl process. For this purpose, the contents of the filter-tube (including the asbestos and glass-wool) are returned to the beaker in which precipitation took place, 20 c.c. of strong sulphuric acid added, the beaker covered with a watch-glass, and heated over a piece of wire-gauze. When frothing has ceased, about 10 grammes of powdered potassium sulphate should be added, and the liquid heated strongly until colourless. It is then allowed to cool, diluted with water, an excess of caustic soda added, the ammonia distilled off, and the distillate titrated with standard acid. The percentage of nitrogen found when multiplied by the factor 6·33 (or in the case of gelatin by 5·5) gives the amount of proteid matter precipitated by bromine.

Experiment showed that the precipitates produced by bromine in solutions of the proteids are insoluble in water and in dilute hydrochloric acid. The extractive matters of meat, on the other hand, are only precipitated in concentrated solutions, and the precipitates formed are, in great part, dissolved on dilution, especially in presence of a little hydrochloric acid.

We have also found that, whilst the precipitates produced by bromine in solutions of the proteids are soluble in dilute caustic soda, the resultant solutions are not completely reprecipitated on acidulation with hydrochloric acid.

In order to further test the process, we have experimented upon the following proteid and gelatinoid bodies:

Commercial Gelatin.—A 10 per cent. solution of commercial gelatin was prepared by dissolving 50 grammes of Nelson's gelatin in warm water and diluting the solution to 500 c.c. In 10 c.c. measure of this solution (containing 1 gramme of gelatin) the nitrogen was determined directly by Gunning's modification of Kjeldahl's process. Another portion of 10 c.c. was diluted, treated with excess of bromine in the manner described above, and the nitrogen in the precipitate determined as before. As these two experiments gave concordant results (14·1 and 14·0 per cent. of nitrogen), we conclude that the bromine precipitated all the gelatin present.

Gelatin-Peptone.—A volume of 300 c.c. of the above gelatin solution was rendered acid by the addition of 8 c.c. of strong hydrochloric acid, and the liquid kept at a

* This is made by placing a little glass-wool in a cylindrical funnel (a vertical glass tube drawn out at the lower end), and covering it with a pad of pulped asbestos. If the filter be properly constructed, no water-pump will be required, and a perfectly clear filtrate will be obtained.

temperature of 100° C., with occasional agitation, until it no longer gelatinized on thorough cooling. It was then made up to its original volume, and the nitrogen determined in the precipitate produced by bromine in 10 c.c. of the solution (= 1 gramme of original gelatin). The result of this experiment gave 13·9 per cent. of nitrogen, which showed that bromine precipitated practically all the gelatin which had been converted into gelatin-peptone, as well as any undecomposed gelatin which might be present. From more recent experiments we are inclined to believe that the conversion of the gelatin was far from complete.

Commercial Egg-Albumin.—A 2 per cent. solution was prepared by dissolving 10 grammes of commercial egg-albumin (in scales) in warm water and diluting the solution to 500 c.c. In 50 c.c. of the solution (= 1 gramme of the original albumin) the nitrogen was determined direct as in the gelatin, the amount found being 8·82 per cent. Another portion of 50 c.c. was treated with bromine, and the nitrogen in the precipitate determined as before, 8·72 per cent. being the result obtained. Hence egg-albumin is completely precipitated by bromine.

Acid Egg-Albumin.—To 100 c.c. of the solution of egg-albumin 2·5 c.c. of strong hydrochloric acid was added, and the liquid kept at 100° C. for several hours, and with frequent agitation. After cooling, the liquid was treated with bromine, and the nitrogen in the precipitate determined. The result showed that the whole of the nitrogen passed into the bromine precipitate. In another experiment a known volume of standard hydrochloric acid was added to another portion of the egg-albumin solution, and the liquid kept at 100° C. for some time, as before. After cooling, such a volume of standard soda was added as would exactly neutralize the hydrochloric acid employed. The nitrogen in the precipitate produced by bromine was determined, and showed in this case, as in that in which when no attempt was made to neutralize the free hydrochloric acid, that the whole of the acid albumin was precipitated by bromine.

Another sample of commercial scale-albumin was converted into acid-albumin (syntonin) by heating it in 1 per cent. solution with hydrochloric acid for six hours. The nitrogen was then determined directly in the resultant liquid and in the bromine precipitate produced in different ways, as shown below. The figures are calculated to 100 parts of the original albumin, which was found to contain 9·88 per cent. of nitrogen and to yield 8·32 per cent. of ash on ignition. Hence the sample was far from pure.

	Nitrogen.
A. By direct Gunning-Kjeldahl process on syntonin solution ...	9·86 per cent.
B. By precipitate from unneutralized syntonin solution	9·76 ,,
C. By precipitate from nearly neutralized syntonin solution ...	9·69 ,,
D. By precipitate from syntonin solution, rendered strongly alkaline and then re-acidified	9·60 ,,
E. Precipitate from syntonin solution, made strongly alkaline by soda, bromine added, and the liquid acidulated after half an hour	6·69 ,,
F. Precipitate from syntonin solution, made strongly alkaline by soda, bromine added, and the liquid acidulated after twenty-four hours	3·52 ,,

From experiments E and F it appears that, in presence of excess of alkali, bromine causes such a change in a solution of syntonin as to prevent full precipitation occurring on subsequent acidulation. The change, which is probably due to the formation of hypobromite and its action on the proteid, appears to be gradual, since the precipitation was far less perfect after twenty-four hours' action than after only half an hour.

Digested Egg-Albumin.—One hundred c.c. of the original egg-albumin solution was acidulated with hydrochloric acid, and digested with pepsin for four hours. The liquid was then cooled, and divided into two equal parts. In one of these the nitrogen was determined directly, and in the other in the precipitate produced by bromine. The results showed that the precipitate formed by bromine in the solution of the mixed products of the digestion of egg-albumin by pepsin contains the total nitrogen of the original albumin.

Albumin-Peptone.—This was prepared by digesting the finely-divided whites of hard-boiled eggs for four hours with pepsin, in presence of free hydrochloric acid. The digested liquid was then raised to a temperature of 97° C. (by blowing in steam) to arrest any further action of the pepsin. The liquid was filtered and neutralized with sodium carbonate solution, and the precipitated syntonin filtered off. The filtrate was evaporated to a low bulk on the water-bath, and was then saturated with zinc sulphate. The precipitated albumoses were washed with a saturated solution of zinc sulphate, and the filtrate and washings diluted to 100 c.c. The total nitrogen was determined directly in one portion of the solution of peptone thus obtained, and the amount of nitrogen in the precipitate produced by bromine in another portion. The results showed that the mixture of peptones resulting from acid-pepsin digestion of egg-albumin is completely precipitated by bromine.

White of Egg.—Another series of experiments on fresh white of egg gave the following results:

	Nitrogen.	= Proteids.
In original white of egg	1·89 per cent.	11 96 per cent.
In precipitate by bromine	1·88 ,,	11·90 ,,
In precipitate by bromine from syntonin solution	1·89 ,,	11·96 ,,

Meat-Extractives.—Solutions of creatinine, asparagine, and aspartic acid were found to yield no precipitate with bromine. To test the powers on the mixture of meat-bases and "extractive matters" actually present in meat-extracts, we allowed some finely-divided raw beef to stand in contact with ten times its weight of water, with occasional agitation, for about six hours. The liquid was strained, and steam blown through the liquid until it had attained a temperature of 97° C. The coagulated albumin was filtered off, and the filtrate was measured. The total nitrogen in it was determined, and also the nitrogen in the small precipitate produced by bromine. In order to determine the latter more exactly, one-fourth of the filtrate from the albumin was evaporated to one-tenth of its bulk and excess of bromine added. The greater part of the precipitate produced dissolved on adding an equal measure of water, and on the addition of a few drops of hydrochloric acid further solution occurred. The remaining precipitate was filtered off and the nitrogen in it determined, the amount found being very trifling.

The following is a tabular view of the chief of the foregoing results :

Substance.	Nitrogen per cent.		Nitrogen multiplied by factor.		Factor employed.
	Total in Original Substance.	Precipitated by Bromine.	Total in Original Substance.	Precipitated by Bromine.	
Commercial gelatin ...	14·10	14·00	77·5	77·0	5·5
Gelatin-peptone	14·10	13·90	77·5	76·5	5·5
Commercial scale albumin	8·80	8·72	55·8	55·2	6·33
Syntonin from scale albumin	9·86	9·76 9·69 9·60	62·41	61·78 61·34 60·77	6·33
Digested scale albumin ...	8·89	8·81	56·3	55·8	6·33
Fresh white of egg ...	1·89	1·88	11·96	11·90	6·33
Syntonin from white of egg	1·89	1·89	11·96	11·96	6·33
Peptone from white of egg	0·70	0·69	4·43	4·37	6·33
Beef-extractives	0·33	0·004	2·11	0·03	6·33

These results fill certain blanks left by the experiments of Rideal and Stewart, and, while generally confirming their conclusions, show that by the very simple mode of operating adopted by us a complete precipitation and determination of proteid and allied matters can be effected, with fairly sharp separation from co-occurring organic matters.

On applying the bromine method to commercial meat-extracts, the following results were obtained. The solutions of the bovril preparations were not previously filtered, and therefore the figures include the nitrogen of the meat-fibre present :

	Nitrogen in Precipitate by Bromine.	× 6·33 = Proteids.
Liebig Company's extract	1·41 per cent.	8·92 per cent.
Seasoned bovril	1·94 ,,	12·28 ,,
Bovril for invalids	2·64 ,,	16·71 ,,

König and Bömer have recently shown that the proteid nitrogen in meat-extracts has generally been much over-estimated. They found a total of 1·17 per cent. of proteid nitrogen in Liebig Company's extract, which is equivalent to 7·41 of total proteids (mostly albumose).

Similarly, a recent analysis of Liebig Company's extract, made by entirely different methods, gave us 9·37 per cent. of total proteids.

The fact that bromine completely precipitates all proteid and gelatinoid matters, affords a convenient means of solving certain problems which have hitherto presented considerable difficulty. Thus, by saturating the liquid with zinc sulphate, and treating the filtrate with bromine, the real peptone formed in digestion-experiments might probably be accurately determined.

We have already applied this process to the vexed question of the existence of real peptones in meat-extracts. Thus, 5 grammes of the Liebig Company's extract was dissolved in 100 c.c. of water, and the solution saturated with zinc sulphate. On adding bromine to the filtered liquid, a precipitate was produced which redissolved on

diluting with water and adding hydrochloric acid. When the filtrate from the zinc sulphate precipitate was diluted with water and acidulated previously to adding bromine, no precipitate was formed. This experiment appears to decide the question of the presence of considerable quantities of real peptones in Liebig's extract in the negative, and hence confirms the conclusion of König and Bömer on this point.

We further propose to examine the applicability of the bromine process of precipitation to the examination of albuminous urine, and to other cases in which a rapid determination of proteid and gelatinoid matters is a desideratum.

ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

FOOD AND DRUGS ANALYSIS.

Distillation of Highly-diluted Ethyl Alcohol (1 in 3,000 to 1 in 10,000). Nicloux and Bauduer. (*Ann. de Chim. Analyt.*, vol. ii. [11], pp. 202-204.)—It has been found by experience that by collecting at the first distillation $\frac{1}{20}$th part of the alcoholic liquid, then beginning again with the original volume and collecting $\frac{2}{20}$, and, from a third distillation, $\frac{3}{20}$, the percentage of alcohol distilling over is in inverse ratio to the concentration of the liquid. For instance, in a mixture of alcohol and water, 1 : 500 – 1 : 1,000, the whole of the alcohol of the former is not over when one-third of the liquid has been drawn over; whereas when the concentration is below 1 : 3,000, the entire volume of the alcohol may safely be considered as contained when the distillate amounts to one-fourth the original quantity. The estimation can then be performed by the Nicloux bichromate method, by adding a solution of potassium bichromate (19 grammes per litre) and 4 or 5 c.c. of pure concentrated H_2SO_4, and comparing the resulting colour with standard colour samples containing known amounts of alcohol. C. S.

Estimation of the Higher Alcohols in Spirits. X. Rocques. (*Ann. de Chim. Analyt.*, vol. ii. [8], pp. 141-146.)—The method employed at the Paris Municipal Laboratory, which consists in heating the alcohol with sulphuric acid and determining the amount of coloration produced, may be rendered more accurate and delicate by the following modifications, in which the aldehydes are first fixed by the employment of an amine: 100 c.c. of the sample, previously distilled and adjusted so as to contain 50 per cent. of alcohol, are boiled gently for an hour with 2 grammes of metaphenylenediamine chloride and a few granules of pumice in a flask surmounted by a reflux condenser, and, after recooling, distilled. To prevent any spray passing over during distillation, the flask is set at an angle of about 45° and connected to a glass coil. Heat should be applied at a rate sufficient to complete the operation in forty minutes, and the condenser must be long enough (40 inches) to cool the distillate to the ordinary temperature. Seventy-five c.c. of the distillate are collected; this volume contains the whole of the alcohol, and consequently indicates 66·7 per cent. on the alcoholimeter.

To apply the sulphuric acid test, 10 c.c. of the distillate are pipetted into a 100 c.c. flask, and 10 c.c. of pure, colorless, monohydric sulphuric acid poured in down the side of the flask. The whole is shaken up and heated for an hour in a brine-bath boiling at 120° C.

A standard solution of 0·667 gramme of isobutylic alcohol per litre of 66·7 per cent. alcohol (*i.e.*, corresponding to 0·5 gramme per litre of 50 per cent. alcohol), is prepared and treated in the same manner, for the purpose of comparing the resulting colorations. This examination is made on the cooled liquids, in a Dubosc colorimeter, the amount of higher alcohols (per litre of 50 per cent. alcohol) in the sample being found and expressed as isobutylic alcohol by the aid of the formula $\frac{500 \times H}{H^1}$, wherein H = the stratum of the standard liquid and H^1 that of the sample. The depth of colour is not exactly proportional to the quantity of the higher alcohols present. The subjoined list shows the ratio between the apparent and actual amount:

Apparent.	Actual.	Apparent.	Actual.
1·125	1·000	0·379	0·400
1·009	0·900	0·255	0·300
0·886	0·800	0·150	0·200
0·760	0·700	0·060	0·100
0·640	0·600	0·019	0·050
0·500	0·500		

The delicacy of the reaction is nearly twice as great (100 : 55) with the above method, employing 66·7 per cent. alcohol, as in the ordinary method with 50 per cent. alcohol. In the colorimetric examination it is advisable to make several readings, *e.g.*, two with a 10 m.m. and two with a 20 m.m. stratum, and strike the mean of the results; the depth of the yellow tinge, with its green fluorescence, is readily appreciable, especially in a 10 m.m. stratum.

Should the proportion of higher alcohols exceed 1 gramme per litre of 50° alcohol—a rare occurrence in practice—the sample should be suitably diluted with pure 66·7 per cent. alcohol after, or by 50 per cent. alcohol before, being distilled. These standards must be accurately maintained, or the colorimetric results will be irregular; but when carried out as prescribed, the method will give comparable and constant, though not absolute, figures. C. S.

On the Occurrence of Alcohol in Milk. H. Weller. (*Forsch. Ber.*, 1897, iv., 206.)—The examination of a sample of milk which had been complained of by the consumer as leaving an unpleasant after-taste gave the following results:

Specific gravity at 15° C.	1·0335.
Fat	3·89 per cent.
Total solids	13·307 ,,
Alcohol (by weight)	0·96 ,,

The milk was derived from a considerable number of cows which were fed on distillery slummage containing 5·90 per cent. of alcohol.

The substance which caused the unpleasant taste could be expelled from the

milk in a current of steam, and separated from the distillate in fine flakes. Its amount was very small. It could also be isolated from the slummage in a similar manner.

From this instance, the author considers that the statement of König (*Nahr. und Genussmittel*, 1893, 249), that when cattle are fed upon fodder containing alcohol the latter does not pass into the milk, is disproved. C. A. M.

Detection of Margarine in Cheese. H. Bremer. (*Forsch. Ber.*, 1897, 51-53.)—By extracting the fat from the cheese with ether, the author considers that impurities are simultaneously extracted, and in Henzold's method, in which the fat is extracted by means of 5 per cent. potash, all the free fatty acids, as well as those in combination with ammonia, are saponified. The method considered the most satisfactory is to shake the finely-divided cheese with water at 20° to 30°, acidified with sulphuric acid (5 : 200), and to apply centrifugal force, when the fat rises to the surface, whilst the albuminoid substances coagulate at the bottom.

In addition to the determination of the usual constants, valuable information may be obtained from the iodine value of the "liquid" fatty acids. In a number of experiments with pure butter-fat, this iodine value did not exceed 95, even when the cows had been fed on maize oil-cake. A sample of Edam cheese yielded a fat, the liquid fatty acids of which had an iodine value of 93·35, whilst the values obtained with two samples of margarine cheese (Rodam and Backstein) were 110·3 and 109·0 respectively. C. A. M.

The Examination of American Lard. Dr. von Raumer. (*Zeit. angew. Chem.*, 1897, 210-215 and 247-254.)—The continually increasing importation to Europe of American lards having high iodine values has made it necessary to alter the generally accepted limit for that value. The usual physical constants all show so much variation in genuine lard that, in the author's opinion, too much reliance must not be placed on them, while he distrusts Becchi's and Welmann's tests.

Schiff's reagent, however, appears to be capable of distinguishing between rancid and over-heated lards and those adulterated with cotton-seed oil, all of which will reduce the silver solution and give the green colour with the molybdic solution. Five c.c. of the melted fat are shaken with 10 c.c. of the reagent and placed in water at 90° C. for two or three minutes. In the author's experiments fresh lard either gave no colour at all or at most a faint rose, which disappeared in about thirty minutes when cold; slightly rancid lards gave a strong colour, which disappeared more slowly, and strongly rancid and over-heated lards gave a pronounced violet, which did not disappear on cooling. On the other hand, beef stearin and beef stearin containing 30 to 40 per cent. of cotton-seed oil showed no coloration.

No. of Tests.	Fat.	Becchi's Test.	Welmann's Test.	Schiff's Reagent.	Length of Reaction with Schiff's Reagent.
2	Lard, rendered in laboratory	—	—	Faint rose	Disappeared on cooling in 30 minutes.
5	Lard, from local butcher	—	—	,,	,, ,,
2	Lard, rendered in laboratory	—	—	Colorless	Colorless.
5	Lard from butcher ...	—	—	Faint trace, rose	Disappeared in 30 minutes.
3	Lard, butcher, ½ year old	—	—	Strong red	Did not disappear.
6	Lard, laboratory rendered, 2 years old, strongly rancid	Very strong	Green, blue, with ammonia	Dark violet	,, ,,
1	Lard, laboratory, overheated	Reaction	,,	Strong red	,, ,,
2	Pig's back fat, rendered in laboratory	—	—	Colorless	Colorless.
1	Beef stearin	—	—	,,	,,
1	Beef stearin, + 33 per cent. cotton-oil	Strong	—	,,	,,
1	Beef stearin, + 40 per cent. cotton-oil	,,	—	,,	,,

The iodine number of the fat and of the so-called liquid fatty acids is considered by the author to be the most reliable test for lard. Attempts were made to concentrate the liquid acids by crystallization from amyl alcohol at 0° and 4° C., but this only raised the iodine value of the soluble portion by about 20. A modification of Muter and De Koningh's lead oleate method (ANALYST, xiv., p. 61) was therefore adopted. From the examination of a large number of samples, it was found that the iodine value of the liquid fatty acids of German lards seldom exceeded 94, the highest being 96·2. The highest figures obtained with American lards of undoubted purity were 66 and 104 for the fat and fatty acids respectively. C. A. M.

A New Method of Determining Caffeine in Coffee. E. Tassily. (*Bull. Soc. Chim.*, 1897, 766-768.)—The powdered coffee is digested with successive portions of boiling water, a litre of liquid being obtained from 10 grammes. The solution is divided into two parts, each representing 5 grammes. These are evaporated to dryness on the water-bath, the residue treated with 1 to 2 c.c. of sulphuric acid (1 : 10), and taken up with boiling water. The caffeine can then be extracted with cold chloroform from the solution made alkaline with ammonia, or with hot chloroform from the residue obtained on evaporating the aqueous solution with sand and magnesia. The former method gives slightly higher results, but in each instance the figures agree well with those obtained by other processes. C. A. M.

Modification of the Thalleioquin Test for Quinine. F. S. Hyde. (*Jour. Amer. Chem. Soc.*, 1897, xix., 331, 332.)—By substituting a clear filtered solution of calcium hypochlorite (bleaching powder) for the bromine or chlorine water the test is rendered more certain, and the green colour more brilliant. After acidulating the

quinine solution with one drop of sulphuric acid (1 : 4), the hypochlorite solution is added until the blue fluorescence just disappears and the solution acquires a faint golden tint. On now adding a few drops of dilute ammonia (1 : 3), a clear emerald green colour should appear. Potassium or sodium hypobromite cannot be used, as the strong alkali tends to precipitate the quinine. C. A. M.

ORGANIC ANALYSIS.

Colour Reactions of Aldehydes and Ketones. (*Zeit. anal. Chem.*, 1897, xxxvi., 369-376.)—Aldehydes and ketones give colour reactions with green fluorescence with the salts of any of the meta-diamines and also with the salts of the ortho- and paradiamines, but without the intense fluorescence. In making the test, a 0·5 to 1·0 per cent. aqueous or alcoholic solution of the chloride of a meta-diamine is added to the aqueous or alcoholic solution of the substance to be tested. The reaction takes place in a few minutes, and reaches its greatest intensity in about two hours. The liquid becomes colorless on the addition of alkali, but the colour is restored on adding acid. Mineral acids weaken the reaction.

The author's experiments indicate that the reaction always occurs when the carbonyl group is not united with a completely substituted hydrocarbon group.

The following table shows the reactions given by various aldehydes and ketones with meta-phenylene-diamine hydrochloride :

Aldehydes of the Fatty Series.

Formaldehyde, H.CHO, bright citron yellow
Acetaldehyde, $CH_3.CHO$, bright yellowish red
Monochlor-acetaldehyde, $CH_2Cl.CHO$, yellow
Isobutylic aldehyde, $(CH_3)_2 : CH.CHO$, bright orange yellow
Valeric aldehyde, $CH_3.(CH_2)_3.CHO$,, ,, ,,
Croton aldehyde, $CH_3.CH : CH.CHO$, brown red
Tiglic aldehyde, $CH_3.CH : C(CH_3).CHO$, brown red
Methyl-ethyl acrolein, $C_2H_5.CH : C(CH_3).CHO$, bright orange yellow
Aldol, $CH_3CH(OH).CH_2.CHO$, bright orange yellow
Furfural, $C_4H_3O.CHO$, bright orange yellow
Glyoxal, CHO.CHO, yellow, fluorescence after one to two hours

Aromatic Aldehydes.

Benzaldehyde, $C_6H_5.CHO$, bright citron yellow
p.-Cuminic aldehyde, $C_6H_4\langle^{C_3H_7}_{CHO}$, bright orange yellow
Anisaldehyde, $C_6H_4\langle^{O.CH_3}_{CHO}$, orange yellow.
o.-oxybenzaldehyde, $C_6H_4\langle^{OH}_{CHO}$, citron yellow

With green fluorescence.

Ketones.

Acetone, $CH_3.CO.CH_3$, yellow red
Mono-chlor-acetone, $CH_2Cl.CO.CH_3$, yellow red
Di-ethyl ketone, $C_2H_5.CO.C_2H_5$, violet red
Methyl propyl ketone, $CH_3.CO.C_3H_7$, brown yellow
Methyl hexyl ketone, $CH_3.CO.C_6H_{13}$, faint green
Methyl nonyl ketone, $CH_3.CO.C_9H_{19}$, brownish yellow

Reactions are not obtained with the following substances:

(*a*) *Aldehydes of the Fatty Series.*—Chloral, butylchloral, paraldehyde, aldehyde ammonia, sulphaldehyde $\left(S\begin{matrix}CH(CH_3).S\\CH(CH_3).S\end{matrix}CH.CH_3\right)$.

(*b*) *Aromatic Aldehydes.*—Nitro-phthal-aldehyde, vanillin.

(*c*) *Ketones.*—Acetophenone, mono-brom-acetophenone, benzylidine-acetone, benzo-phenone, naphthyl-phenyl-ketone, trioxy-benzophenone, benzoin, phenyl-thionyl-ketone.

The author has also examined the behaviour of Schiff's reagent, and finds that, contrary to the experience of others, it cannot be relied upon when used with a hot solution, since the reagent by itself always shows a distinct bluish-violet colour when cooled after having been boiled; and hence the appearance of a faint colour after boiling cannot be taken as conclusive of the presence of aldehydes or ketones.

On the other hand, a solution of grape-sugar boiled with the reagent and cooled gives no coloration, and from this the author confirms Nickel's observation that grape-sugar exercises a disturbing influence. C. A. M.

On the Determination of Copper as Oxide in the Gravimetric Estimation of Sugar. K. Farnsteiner. (*Forsch. Ber.*, 1897, iv., 169-171.)—The author defends his method (ANALYST, xx., 258) from certain criticisms of Bremer as to its accuracy (*Forsch. Ber.*, 1897, 110), and describes experiments by different chemists with the object of proving that the variations in the reduction method are just as great as in his oxidation process.

In working the reduction method, he has obtained most satisfactory results by substituting methyl alcohol for hydrogen. A moderate stream of dried carbon dioxide is passed through methyl alcohol at the ordinary temperature and then through the tube, which requires rather more heating than when hydrogen is used. When reduction is complete, the tube is allowed to cool in a current of carbon dioxide.

C. A. M.

An Iodometric Method of Estimating Sugar. G. Romijn. (*Zeit. anal. Chem.*, 1897, xxxvi., 349-359.)—When glucose in alkaline solution is acted upon by iodine, gluconic acid is produced in accordance with the equation

$$CH_2OH.(CHOH)_4.CHO + 2I + 3NaOH = CH_2OH.(CHOH)_4.COONa + 2NaI + 2H_2O;$$

but where caustic alkali is used, the gluconic acid is liable to undergo further decomposition. When the caustic alkali is replaced by certain alkaline salts, the results are much more in accordance with theory, and of these salts the most suitable is borax. The temperature, quantity of sugar, and time of action must all be taken into account.

The sugar solution (about 10 c.c.) is placed in a stoppered flask with 25 c.c. of iodine solution, containing 1 gramme of borax and as much iodine as requires, after the addition of acid, from 30 c.c. to 33 c.c. of decinormal thiosulphate for decolorization. The flask is then maintained at 25° C. by means of a thermostat for the

required time, at the end of which 1·5 c.c. of hydrochloric acid (specific gravity 1·126) is added, and the excess of iodine titrated with decinormal thiosulphate.

The results obtained with glucose solutions when the volume of the mixed solutions was 35 c.c. to 50 c.c., the time sixteen to twenty-two hours, and the amount of glucose 154 to 164 milligrammes, showed 97·76 to 99·47 per cent. of the theoretical quantity. With further dilution the results were too high, owing to the hydrolytic dissociation of the borax.

Similar experiments were made with solutions of fructose, which was hardly attacked at all, the figures indicating only about 3 per cent. of the amount taken.

A few of the results obtained with pure specimens of other sugars are given below. In each case 2 atoms of iodine were taken as equivalent to 1 molecule of the sugar. It is noticeable that the aldoses—galactose, mannose, arabinose, xylose, and rhamnose—behaved in the main like glucose.

Sugar.	Quantity in milligrammes.	Hours.	Milligrammes found.	Per cent.
Galactose	169·3	20	171·92	101·55
Mannose	166·4	18½	168·94	101·53
,, ...	155·2	18½	161·89	104·31
Arabinose	149·4	18	144·53	96·75
Xylose	142·7	18½	143·79	100·76
Rhamnose	37·1	18	41·72	112·45
,, ...	146·2	18	145·69	99·65
Chitosamin – HCl .	187·4	18½	199·72	106·56
Sorbose	166·3	18½	4·43	2·66
Maltose	338·4	20	351·94	103·71
Lactose	150·35	17½	169·46	112·72
,, ...	333·55	18	349·04	104·65
Saccharose	334·72	17½	50·67	15·14
,, ...	334·72	17½	51·01	15·24
Raffinose	298·1	18½	122·61	41·13
,, ...	504·5	18½	161·95	32·10
Stachyose	318·6	18½	133·11	41·78

Where the results are too high (*e.g.*, maltose and lactose), it is only necessary to increase the quantity of the sugar taken to obtain the correct amount.

The paper also contains an account of some experiments on other substances. Ethyl alcohol, acetone, and formic acid exercised a strong reducing action on the borax iodine solution; glycerin, lactic acid, and mannite did the same, but in a much less marked degree; whilst acetaldehyde and tartaric acid were practically without action on it. With regard to the determination of mixtures, the author gives a long table in which it is proved that a mixture of glucose and fructose may be estimated as though pure glucose. Thus, 153·78 milligrammes of glucose with 80 milligrammes of fructose yielded 154·21 milligrammes = 100·28 per cent. of glucose. Mixtures of lactose and saccharose gave results about 6 per cent. too high, whilst those obtained with a solution containing glucose and mannite were about 20 per cent. too low. In mixtures of cane-sugar and lactose the figures, which were constant in duplicate determinations, were about 5 per cent. too high.

The author's general conclusion is that this method promises an accurate means of determining aldoses by themselves and in the presence of ketoses and other substances, but that it is not always applicable in practice. C. A. M.

The Insoluble Carbohydrates of Wheat. H. C. Sherman. (*Journ. Amer. Chem. Soc.*, 1897, xix., 291-316.)—I. DESCRIPTIVE.—It is shown that the hemicellulose of wheat apparently yields only pentoses on hydrolysis, and therefore consists of pentose anhydrides, or true pentosans.

The fibre left after treatment with sulphuric acid yields furfural, gives the magenta colour (produced by lignin) on treatment with chlorine and boiling with sodium sulphite, and the red colour characteristic of pentosans with phloroglucinol. When immersed in a solution containing molecular proportions of ferric chloride and potassium ferricyanide it fixes a certain amount of the ferricyanide—a reaction shown by Cross and Bevan to be characteristic of lignocellulose, of which jute is the type.

Lignone chloride ($C_{19}H_{18}Cl_{14}O_9$) isolated by Cross and Bevan from chlorinated jute-fibre, was also obtained from wheat, and it is suggested that this type of lignification may be common in plants of annual growth.

The furfural-producing constituents are the hemicellulose, and bodies in combination in the lignin group (about 40 per cent.), and in the cellulose obtained by the chlorination method (about 10 per cent.); but in the case of wheat these are all of a pentosan nature. The author considers it advisable to distinguish between these, and to group them into those dissolved readily by dilute acids (pentosans proper) and those which resist acid or both acid and alkalies (combined pentosans).

II. ANALYTICAL.—The following scheme gives an outline of the method of analysis proposed. Five grammes of the sample (previously extracted with ether) are stirred with 100 c.c. water for a few minutes, filtered, and washed.

FILTRATE.—Boiled with $\frac{1}{10}$ its volume of 25 per cent. HCl, clarified, and dextrose determined with Fehling's solution in an aliquot portion. Result calculated to	RESIDUE.—Treated with malt-extract till starch disappears, cooled, filtered, and washed.			
	FILTRATE.—treated as in the case of preceding filtrate. Dextrose found calculated to	RESIDUE.—Boiled thirty minutes with H_2SO_4 (1·25 per cent.), filtered, washed with water and alcohol.		
		FILTRATE.—H_2SO_4 to make 2 per cent. added. Boiled under reflux condenser six hours. Reducing power determined and calculated to pentose and thence to	RESIDUE.—Dried and weighed, proteid and ash determined in duplicate residue, and correction made. Treated according to method of Cross and Bevan, dried, and residue weighed.	
			Loss of weight corrected for proteids and ash equals	RESIDUE, less proteid (if present), and ash equals
Soluble carbohydrates as dextrin.	Starch.	Free pentosans.	Lignin and allied substances.	Cellulose.

Starch.—Reference is made to a previous paper by the author (see ANALYST, this vol., 19.), in which the superiority of the malt-extract method over other methods is proved.

True Pentosans.—Sulphuric acid was chosen, as it was found to cause but little alteration in the pentosans even on long boiling. The copper reduced was determined by Allihn's method, and the pentose taken as 97 per cent. of the indicated dextrose. Pentose × 0·88 = pentosan.

Cellulose.—Comparative experiments were made with three methods:

(1) Schulze's method—digesting substance with nitric acid and potassium chlorate, and subsequently washing with 2 per cent. ammonia.

(2) Cross and Bevan's method, in which 30 grammes of fibre are boiled for thirty minutes with 800 c.c. of 1 per cent. sodium hydrate, well washed, squeezed as free from water as possible, and subjected to the action of chlorine for an hour, with occasional stirring. The mass is then filtered, washed free from acid, heated to boiling with 600 c.c. of 2 per cent. sodium sulphite, and sufficient sodium hydrate solution added to make $\frac{1}{5}$ per cent. of the whole. After being boiled for five minutes the solution is filtered hot through a muslin filter, washed till neutral and colorless with water, then washed with alcohol, dried and weighed.

(3) Lange's method—fusion with alkali (ANALYST, xx., 283).

The results obtained by the three methods, as also by exposure to chlorine, wet, and without treatment with alkali, showed the superiority of Cross and Bevan's method.

	Description of Product.				
Method	Yield per cent.	Furfural per cent.	Nitrogen per cent.	Ferricyanide fixed, per cent.	Colour.
Cross and Bevan ...	66·5	5·62	0·00	9·20	Light cream.
Schulze	66·0	7·00	0·22	6·04	Light brown.
Lange	41·2	3·96	0·03	0·89	Nearly white.
Chlorination alone ...	84·3	8·89	0·08	2·02	Very light brown.
Original sample ...	100·0	11·46	0·25	11·17	

Determination of Furfural.—For this a modification of the method of the Association of Official Agricultural Chemists is used. From 2 to 5 grammes are distilled with 100 c.c. of hydrochloric acid (specific gravity 1·06), each 30 c.c. passing over being replaced by the hydrochloric acid, and the distillation continued until the distillate gives no red colour with aniline acetate. The distillate is made up to 400 c.c., and for each 50 c.c. of water used 10·2 grammes of sodium chloride are added. The liquid is exactly neutralized with sodium carbonate, and 10 c.c. of a solution of phenylhydrazine acetate added. This is prepared by mixing 12 grammes of phenylhydrazine with 7·5 grammes of glacial acetic acid and diluting to 100 c.c. After being well stirred for thirty minutes the whole is kept in the dark until next day. The precipitate is collected on a glass-wool filter, and dried to constant weight at 55° to 60° in a slow current of air. It is then dissolved off the filter with alcohol, the filter-tube dried and weighed, and the loss of weight calculated as furfural hydrazone. From the result the pentosans can be calculated by the factors

Hydrazone × 0·516 + 0·0104 = furfural.
Furfural × 1·84 = pentosans.

III. Physiological.—The author was fortunately able to obtain samples of the food and fæces of a steer, which was being fed for experimental purposes on wheat-bran only, and to ascertain the digestibility of each of the constituents as determined by this method. The following are the results calculated on the dry substance:

	Food per cent.	Fæces per cent.	Per cent. digested.
Soluble carbohydrates as dextrin	7·2	0·7	96·9
Starch	17·7	0·0	100·0
Free pentosans	17·5	18·7	66·2
Cellulose	8·5	20·2	24 8
Lignin and allied substances	11·6	23·2	36·7
Protein	20·49	11·04	82·96
Ether extract	6·92	12·52	42·73
Ash	6·05	11·04	42·21
Undetermined	4·04	2·60	...
Nitrogen-free extract...	55·59	41·93	76·08
Crude fibre	10·96	23·47	32·21

The total dry matter digested was 68·33 per cent. of the whole. These figures prove that the digestibility of the insoluble carbohydrates ranges from 25 to 100 per cent., and since there is also a possible difference in the physiological value of the digested portions, a distinction between starch and pentosans should always be made in food analysis. While the normal carbohydrates are known to be completely digested in the healthy body, it has been held that lignin is but little oxidized, and supplies directly the benzol radicle in the hippuric acid in the urine of herbivorous animals (Stutzer, *Ber.*, viii., 575; Weiske, *Zeit. Biol.*, xii., 24). With regard to the pentoses and pentosans authorities differ. Ebstein (*Arch. path. Anat.*, cxxix., 401) came to the conclusion that they were practically without nutritive value. Salkowski (*Cent. med. Wissen.*, 1893, 193) and Cramer (*Zeit. Biol.*, xix., 484) found that they were digested for the most part, the latter holding that they caused an increased storage of glycogen in the liver. On the other hand, Fretzel (*Archiv. ges. Physiol.*, lvi., 273), from experiments on rabbits with xylose, concluded that in no way did this sugar contribute to the formation of glycogen. C. A. M.

Estimation of Glycerol. C. Böttinger. (*Chem. Zeit.*, 1897, xxi., 658.)—The author has attempted to work out a process based on the conversion of the glycerol into its triacetic ester by means of potassium bisulphate and acetic anhydride. Under the conditions detailed in the paper he has succeeded in obtaining from commercial glycerin dehydrated at 170° C. a fairly uniform yield, averaging in twelve experiments 151·4 per cent. (maximum 157·4 per cent., minimum 143·6 per cent.), whereas theory demands 237 per cent.

As regards methods depending on the treatment of alcoholic wine extracts with ether, he finds on acetylating the different products (1) that nearly half such an extract is soluble in the ethereal liquid, (2) that the soluble portion is by no means pure glycerol, but may contain 43 per cent. of other substances, and (3) that the undissolved part of the extract may retain even more glycerol than is actually taken up by the ether. F. H. L.

The Analysis of Commercial Xylidine. W. Vaubel. (*Zeit. anal. Chem.*, 1897, xxxvi., 285-289.)—Commercial xylidine usually contains the five isomerides—α-and-β-ortho-xylidine, α-and-β-meta-xylidine, and para-xylidine, of which the chief are the α-meta compound, or asymmetric meta-xylidine and para-xylidine.

To determine the amount of the former, advantage is taken of the fact that its acetate and chloride are insoluble in acetic acid and hydrochloric acid respectively, the most satisfactory results being obtained with acetic acid. A hundred grammes of the substance are mixed with 100 grammes of glacial acetic acid, and after twenty-four hours the precipitate is filtered off with the aid of a suction-pump, air-dried, and weighed. It consists for the most part of the acetate of asymmetric meta-xylidine, but is liable to contain a small amount of the acetyl derivative, or of the acetate of one or more of the other isomerides.

On treatment with a solution of bromine in hot sodium hydrate the two meta-xylidines absorb 1 atom of bromine, as do also their acetyl derivatives, whilst the other isomerides absorb 2 atoms. But as the acetate of β-meta-xylidine is very soluble in acetic acid, only traces of it can occur in the precipitate.

The amount of the acetyl derivative in the precipitate can be determined by the rate at which the bromine is absorbed, for it absorbs its 1 atom of bromine much more slowly than does the salt. It is best to hydrolyse it before the titration by boiling for ten minutes with hydrochloric acid.

In illustration of the method, the following results of the analyses of a technical xylidine are given :

Specific gravity at 20° C. = 0·982

Fractional distillation (750 m.m.)	210°	211°	212°	213°	214°	215°	residue
Per cent.	14	18	18	24	6	2	2

A hundred grammes of the xylidine, mixed with 100 grammes of glacial acetic acid, yielded 32 grammes of solid acetate, corresponding to 20·8 per cent. of α-meta-xylidine. But on treatment with the bromine solution, it was found that about half of the precipitate consisted of the acetyl derivative. The bromine required for 16 grammes of acetate = 7·1 grammes, and for 16 grammes of the acetyl derivative = 7·8 grammes—together = 14·9 grammes. But the actual amount of bromine absorbed was 17·9 grammes; therefore the 20·8 grammes of precipitate contained 3·5 grammes of the acetate of ortho- or of para- xylidine, since these can absorb 2 atoms of bromine.

C. A. M.

Colour Reactions of Nitric and Chloric Acids with Certain Aromatic Bodies. E. C. Woodruff. (*Journ. Amer. Chem. Soc.*, 1897, xix., 156-169.)—This paper gives the particulars of upwards of 300 reactions of different inorganic salts, with the various types of aromatic substances. In general, chloric acid produces a darker colour than nitric acid, and although many of the tests will detect them separately, in most cases, when mixed, the effect of the nitrate is completely masked.

A modification of the phenol test for nitrates is suggested; it is said to be more delicate than the usual test. One c.c. of concentrated sulphuric acid is placed in a test-tube, and on it is floated a layer of not more than 0·5 c.c. of a saturated aqueous solution of phenol. A drop of the unknown solution is now allowed to fall upon the

upper layer, generally producing a brown ring. After standing for some time the whole is shaken and neutralized, when a picrate-coloured solution indicates a nitrate. Chlorates produce a brownish-yellow, instead of a greenish-yellow, colour.

Diphenylamine (4 per cent. solution) is a delicate test for nitrates, but has the disadvantages that it will not keep well in solution, that it is not possible to dilute or neutralize it so as to eliminate the effects of bromides and iodides, that nitrates, chlorates, bromates, and iodates all give practically the same colour with it, and that it must be kept cool. The most satisfactory reagent for detecting nitrates in the presence of chlorates and other inorganic salts is a mixture of dimethylaniline (2 drops), and paratoluidine (0·2 gramme) in sulphuric acid (10 c.c.). This gives a blood-red colour with nitrates if the chlorate is not present in too great excess.

Two methods of removing or reducing the strength of chlorates found satisfactory are:

(1) The solution is acidified with sulphuric acid and boiled, after the addition of hydrochloric acid, until on adding another drop of hydrochloric acid no yellow colour appears. The phenol test will now show the nitrates without the slightest trace of brown due to the chlorate.

(2) The solution is evaporated just to dryness, and water added in sufficient quantity to dissolve the nitrate, but not all the chlorate, which is much less soluble. The solution is decanted, diluted, and examined by the dimethylaniline test.

But the best method of applying the test is to add to the unknown solution a little potassium chlorate, if not already present, and then to boil with hydrochloric acid. This oxidizes all reducing agents that might interfere with the test without introducing enough chlorate to mask the nitrate reaction. C. A. M.

Examination of Sumach. M. Spica. (*Gazz. Chim. Ital.*, 1897, xxvii., 349; through *Chem. Zeit. Rep.*, 1897, 173.)—Sumach is frequently adulterated with the leaves of *Tamarix Africana* (Sicilian "bruca,") or of *Pistacia lentiscus* ("stinco"). Five grammes of the samples are boiled for half an hour in 500 c.c. of water, the loss by evaporation made up and the decoction filtered. Twenty-five c.c. are shaken with 5 c.c. of a solution containing 250 grammes of basic lead acetate in 1 litre of water (specific gravity 1·184) and 15 c.c. of caustic potash (specific gravity 1·155), and the mixture concentrated to a volume of 15 c.c. The liquid should be quite clear, any precipitate indicating impurity or adulteration. It is then diluted to 250 c.c., filtered, and examined colorimetrically, when the depth of tint should be equal to that of a solution of 0·15 gramme of safranine in 1 litre of water; any foreign matter causes the liquid to be paler in shade.

The nitrogen in sumach is 0·912 per cent., in tamarix 1·77 per cent., and in pistacia 1·63 per cent.; while the amounts of ash are 6·60, 12·40 and 5·40 per cent. respectively. F. H. L.

Betanaphthalenesulphonic Acid as a Reagent for Albumin, Albumoses and Peptones. E. Riegler. (*Pharm. Central H.*, 1897, xxxviii., 379; through *Chem. Zeit. Rep.*, 1897, 173.)—Five or six c.c. of the liquid under examination are mixed in a test-tube with 20 or 30 drops of a filtered 5 per cent. solution of β-naphthalene-

sulphonic acid in water. One part of albumin in 40,000 of water gives a noticeable precipitate which does not disappear on heating. Albumoses and peptones yield precipitates which dissolve on warming, but reappear when the liquid becomes cold again. F. H. L.

Analysis of Commercial Albumin. P. Carles. (*Jour. Pharm. Chim.*, 1897, 102-104.)—It is not uncommon to find samples of albumin containing from 12 to 25 per cent. of insoluble coagulated matter without the power of fining wine, whilst others contain gum, dextrin, or gelatin.

Two grammes of a preparation are treated with distilled water up to 200 c.c. If the albumin is free from coagulated matter, the solution is quite transparent. To 100 c.c. of the solution, 35 c.c. of a 1 per cent. of *pure* tannin are added, and about 0·2 gramme of powdered potassium bitartrate. The liquid is well shaken, and a small portion filtered. One portion of the filtrate is tested for excess of tannin, and to another portion several drops of the tannin solution are added.

If tannin be found, it proves that of the 0·35 gramme added originally, part has not been taken up by the albumin, which is accordingly deficient in true albumin, and contains either inert substances, fraudulently added, or overheated portions.

If, on the other hand, the addition of more tannin solution causes a turbidity, the inference is that the albumin contains a substance precipitable by tannin, and which has for tannin a greater saturation capacity than the albumin itself. All the gelatins have the property of precipitating about four times as much tannin as desiccated albumin.

As a further test, 100 c.c. of the solution are warmed on the water-bath in order to coagulate all the albumin present. After filtration of a heated solution of pure albumin, no further precipitate with tannin is obtained, whereas there is an abundant one when gelatin is present. In the same filtered liquid, dextrin, gums, and other substances, if present, may be detected by the usual methods. C. A. M.

Estimation of Gelatin. P. Carles. (*Ann. de Chim. Analyt.*, vol. ii. [10], pp. 181, 182.)—The author believes that the method proposed by Jean (*Analyst*, this volume, p. 164), though excellent for a gelatin liquor of uniform character, and one wherein all the glue constituents are completely gelatinized, would give rise to error if used on a bouillon in course of manufacture, owing to the differences in the time of boiling necessary for the conversion of the various albuminoids, as well as to the differences in the properties of the resulting products.

The precipitating action of the commercial gelatins on pure tannin is very divergent, 10 grammes of Russian fish-glue, dissolved at 40° C., throwing down 4 grammes of tannin, whilst the same quantity of granulated extra gelatin precipitates 8 grammes; cut fish-glue from 3 to 10 grammes (according to the state of division or solution); bone-gelatin and brown Givet glue 11 grammes; Bordeaux gelatin 11·50 grammes; and Laîné gelatin 12·50 grammes of tannin. C. S.

Optical Analysis of Urine, Positive and Negative Thermo-optical Diabetic Sugar. Fr. Landolph. (*Rev. de Chim. Analyt. Appl.*, vol. v. [14], pp. 278, 279.)—

Thermo-optically Positive (true diabetic) Sugar.—The principal characteristics of this sugar are: stability, rapidity of fermentation—about thirty minutes at 15° C. and under—and high reducing power, the latter being double that of grape sugar, *i.e.*, 100 grammes correspond to 441 grammes of copper oxide. When examined by the polaristrobometer at the ordinary temperature, and again after boiling, the optical power is found to increase, *i.e.*, the sugar is thermo-optically positive. The results of this examination agree with those obtained by determining the carbon dioxide evolved during fermentation; but the reduction method with Fehling's solution is inaccurate, being based on the erroneous assumption that this sugar has the same reducing power as grape sugar. The precipitate of hydrated cuprous oxide formed by diabetic sugar is yellow and more voluminous than the anhydrous red oxide thrown down by grape sugar, uric acid, etc., but the latter oxide is sometimes furnished by diabetic urines that have undergone transformation.

Thermo-optically Negative Sugar.—This always accompanies diabetic sugar, but, like grape sugar, loses its positive rotatory power when boiled. It ferments more slowly than true diabetic sugar, and is generally found anterior to diabetes. The author has frequently examined urines exhibiting a dextrorotation of several degrees and shown, both by fermentation and by the polaristrobometer, to contain sugar, but which on boiling gave only the normal levo-rotation due to free uric acid and urates.

C. S.

Rapid Estimation of Uric Acid in Urine. E. H. Bartley. (*Jour. Amer. Chem. Soc.*, 1897, xix., 649-656.)—The method is based on the complete precipitation of uric acid by silver nitrate in the presence of excess of magnesia mixture and ammonia, the end-point being detected by an alkaline sulphide. The precipitation of the xanthine bases by the silver nitrate is obviated by titrating in a hot solution.

To 50 or 100 c.c. of clear urine are added 5 c.c. of magnesia mixture and about 10 c.c. of ammonium hydrate (specific gravity, 0·960). After warming the mixture on the water-bath, $\frac{1}{60}$ N. silver nitrate is run in, until a drop removed by means of a pipette with cotton tied over the end, to act as a filter, gives a dark coloration when brought into contact with a weak solution of sodium sulphide. Experiments with pure water showed that 0·5 c.c. of the silver solution in 50 c.c. was required to give a marked reading, and this amount must therefore be deducted from the reading.

Each c.c. of silver nitrate corresponds to 0·00336 gramme of uric acid, and from the number of c.c. used (less 0·5 c.c. for each 50 c.c. of urine), the amount of uric acid can be calculated.

If, after the titration is complete, the solution is cooled, an addition of 1 to 3 c.c. of the silver solution will be required to again produce the end-reaction, the xanthine bases being precipitated by the silver in a cold solution.

The results obtained in this way, as compared with Ludwig's method, which has been proved to give too low results, were:

		Ludwig. Gramme.		Direct Titration. Gramme.
No. 1.	Uric acid in 100 c.c. ...	0·0822	...	0·08064
No. 2.	,, ,, ,, ...	0·0506	...	0·05374
No. 3.	,, ,, ,, ...	0·0684	...	0·07064

C. A. M.

Lintner's Reaction of Guaiacum with Diastase. **B. Pawlewski.** (*Ber.*, 1897, xxx., 1813.)—The blue colour produced by an alcoholic solution of guaiacum mixed with hydrogen peroxide in presence of active diastase is only characteristic of the latter if the reaction occurs instantaneously, and if there is no other body at hand capable of acting in a similar manner. Should the colour only appear after a few minutes the test is valueless, for many substances will determine it; in fact, guaiacum tincture gives a blue colour with hydrogen peroxide alone, especially on warming.

F. H. L.

Improvements in Ultimate Organic Analysis. **M. Dennstedt.** (*Ber.*, 1897, xxx., 1590.)—This process effects the simultaneous estimation of N, S, and Cl (or other halogen), as well as the C and H in any organic body. The combustion tube is about 83 cm. long, open at both ends, and is heated by a number of flat-flame burners rather than in the ordinary furnace. At the centre it contains a layer 6 or 8 cm. long of platinum black, prepared by igniting some such compound as the chloroplatinate of pyridine, which, provided a proper current of oxygen is constantly maintained, need only be raised to a dull red heat to ensure complete oxidation. At the further end of the tube the usual apparatus for absorbing the water and the carbon dioxide are provided.

If the substance contains nitrogen, NO_2 is produced, and this is absorbed as such in two boats filled with lead peroxide placed in the tube behind the platinum black, and heated to about 150° by means of a special burner, the boats being weighed before and after the operation. If the body contains only Cl,* two boats of precipitated silver are employed at a temperature of 200° or 300°, and weighed as above. If N, as well as Cl, is present, both the lead and the silver boats are inserted in the tube; but only the latter require weighing. If the substance contains only S,* all four boats are used, and all must be weighed. The greater part—often the whole—of the sulphur is oxidized to sulphuric acid, and is directly absorbed by the silver; some is only burnt to sulphurous acid, and is taken up by the lead as SO_2. In presence of both S and N, the contents of the lead boats are extracted with 33 per cent. alcohol, and a portion of the filtrate evaporated to give the amount of lead nitrate formed; this is calculated into NO_2, when the remainder of the increase in weight represents the SO_2. If both S and Cl occur, the silver chloride and sulphate are separated by being dissolved in potassium cyanide, filtered, the HCN displaced by HCl, the silver chloride removed, and the sulphuric acid in the filtrate precipitated with barium. The weight of the SO_4 so obtained, deducted from the total gain of the silver boats, gives the amount of halogen. In the event of N, Cl, and S being all present, the lead oxide boats absorb the N as NO_2 and $\frac{1}{2}$ or $\frac{1}{3}$ of the S as SO_2, and the silver boats take up the Cl or other halogen and the remainder of the S as SO_4.

The author states that the results are quite accurate, and he appends the figures derived from the actual analysis of parabromphenylthiourea, which agree well with those demanded by theory.

F. H. L

* Beside the C, H, and O.

Yellow Light for Saccharimeters. F. Dupont. (*Rev. de Chim. Analyt. Appl.*, vol. v. [10], p. 186.)—The author finds that a fused mixture of chloride and tribasic phosphate of sodium in about molecular proportions furnishes a salt which fuses more readily than the chloride, and does not decrepitate, thus giving a steadier and clearer flame than that obtained with sodium chloride alone. C. S.

INORGANIC ANALYSIS.

The Separation of Molybdenum and Tungsten. E. D. Desi. (*Jour. Amer. Chem. Soc.*, 1897, xix., 242.)—At the conclusion of a long communication on the oxides of tungsten, the author refers to the methods of separating molybdenum and tungsten. Rose's method, in which the molybdenum is precipitated from the mixed solution as sulphide by adding tartaric acid and passing in hydrogen sulphide to saturation was shown by Friedheim and Muller (*Zeit. anorg. Chem.*, i., 76) to be not quite satisfactory, a small quantity of molybdenum always remaining in solution. The fact that tungstic acid is completely insoluble in concentrated or dilute sulphuric acid, hot or cold, whereas molybdenum trioxide is readily soluble, affords a simple and exact method of separating the two elements. C. A. M.

Separation of Nickel from Cobalt, Nickel from Iron, and Cobalt from Aluminium. E. Pinérua. (*Ann. de Chim. Analyt.*, vol. ii. [11], pp. 201, 202.)—The method proposed is based on the insolubility of the chlorides of nickel and aluminium in ether saturated with hydrochloric acid, in which reagent cobalt and iron are readily soluble. The metallic chlorides in question are dissolved in a small quantity of water, and after adding 10 to 12 c.c. of fuming hydrochloric acid and 10 c.c. of (preferably anhydrous) ether the liquid is agitated and saturated with gaseous hydrochloric acid at a low temperature. Nickel is thrown down as a heavy crystalline yellow chloride which is washed on the filter with ether saturated with gaseous hydrochloric acid, and the metal determined by any of the usual methods, *e.g.*, as sulphate. If iron be present along with cobalt the filtrate will be greenish-blue or yellow-green.

Aluminium and cobalt are separated in the same way, the former being precipitated as chloride. In separating nickel from iron the nickel precipitate must be repeatedly washed with ether, and, if necessary, redissolved and the operation repeated.

From the behaviour of the commercial chlorides of nickel and cobalt in presence of solvents the author shares the belief of Winckler and others that these metals are unknown in a pure state, but probably contain hitherto unidentified elements.

C. S.

Separation of Iron and Alumina by the Aid of Sodium Peroxide. C. Glaser. (*Chem. Zeit.*, 1897, xxi., 678.)—The solution is nearly neutralized with ammonia and dry sodium peroxide (2 to 3 grammes of Na_2O_2 to 0·4 gramme of substance) added until the liquid becomes clear and all the iron is converted into ferrate. The whole

is boiled, allowed to settle, and filtered. The precipitate of ferric hydrate is dissolved in acid and thrown down again by means of ammonium sulphide from a solution containing ammonium citrate or tartrate; while the filtrate is acidified and the alumina determined by phosphoric acid and ammonia acetate, or any of the usual methods.

The original liquid should be freed from silica, and although the iron precipitate does not carry down any phosphoric acid with it, but is contaminated merely with adhering alkali, any considerable amount of calcium phosphate should be removed with ammonium acetate or by Glaser's process before the addition of the sodium peroxide. F. H. L.

The Determination of Sulphur in Pig-iron. A. A. Blair. (*Jour. Amer. Chem. Soc.*, 1897, xix., 114, 115.)—The author's experiments prove that sulphur may be present in pig-iron in four forms: (1) Sulphur evolved as hydrogen sulphide on dissolving the iron in hydrochloric acid; (2) Sulphur evolved as other compounds, not absorbed by alkaline lead-salts or oxidized by bromine or potassium permanganate; (3) Sulphur unacted on by boiling hydrochloric acid, but oxidized by nitric acid or *aqua regia*; (4) Sulphur unacted on by nitric acid, hydrochloric acid, or *aqua regia*.

Bamber's method (*Jour. Iron and Steel Inst.*, 1894, i., 319) is recommended for treatment of pig-irons containing sulphur in the last form. Five grammes are dissolved in strong nitric acid, 2 to 5 grammes of potassium nitrate added, and the solution evaporated to dryness in a platinum capsule and ignited. The residue is treated with water containing a little sodium carbonate, filtered, and the filter washed also with water containing sodium carbonate. The solution is acidulated with hydrochloric acid, evaporated to dryness, redissolved in water containing a few drops of hydrochloric acid, brought to the boil, and the sulphur precipitated with barium chloride. C. A. M.

The Composition of Red Lead. D. Woodman. (*Jour. Amer. Chem. Soc.*, 1897, xix., 339-341.)—For certain purposes, it is important to know the degree of oxidation in specimens of red lead, and this the author determines by digesting the red lead in a saturated solution of normal lead acetate, which dissolves the litharge in excess of that in combination as Pb_3O_4. In this way a series of results was obtained showing a variation in the amount of actual red lead of from 41 to 92 per cent. The small amounts of silica, lime, oxide of iron, and carbon dioxide normal in the commercial article are neglected:

Red lead ...	Pb_3O_4	51·0	56·0	75·0	41·0	90·0	66·0	87·5
Litharge ...	PbO	49·0	44·0	25·0	59·0	10·0	34·0	12·5

The author quotes from Gmelin the figures showing the amount of red lead formed from massicot. One firing of twenty-four hours in a reverberatory furnace yielded 50·0 per cent. of red lead; two firings yielded 52·1; five firings, 66·2 per cent.; and eight firings, 74·8 per cent. Red lead from "white lead," after three firings of twenty-four hours each, contained 95·3 per cent. Hence these results seem to show that there has been little or no improvement in the methods of manufacture, at least as regards more complete oxidation. C. A. M.

APPARATUS.

A New Hot-Water Oven for Temperatures over 100° C. F. Gantter. (*Forsch. Ber.*, 1897, iv., 154, 155.)—This is a double-walled oven with an opening for a thermometer. The jacket is filled full of water, and the outlet through which steam usually escapes is connected with a barometer tube containing mercury. By regulating the gas-flame any temperature, from 100° to 110° or higher, corresponding to the pressure, may easily be maintained. Near the bottom of the oven there is a safety-valve. C. A. M.

REVIEW.

THE ANALYST'S LABORATORY COMPANION. BY ALFRED E. JOHNSON, A.R.C.S.I., F.I.C. Second edition (J. and A. Churchill). Price 5s.

This admirable and handy little volume, which contains all the tables usually required by analysts, has been carefully revised and has now reached its second edition. A new table of five-figure logarithms takes the place of the former seven-figured one, a saving of time being effected by its use without the sacrifice of too much accuracy. The section on weights and measures has been entirely re-written, and the tables dealing with the specific rotatory and reducing powers of the carbohydrates extended in the light of recent research. The nitrogen to albuminoid matter table has been re-calculated on the basis of the more modern factor 6·25 instead of the older one of 6·33. A new and useful table for calculating c.cs. of pentinormal acid into their equivalents of nitrogen, for use in the Kjeldahl process, has been introduced. Improvements have been made in the alkali tables, and also some new and useful matter inserted in connection with alcohol estimation. These are *some* of the novelties and improvements met with, and they serve to show that the work has been fully brought up to date. The little useful facts and hints interspersed throughout the work constitute not the least valuable portion of it. Blank pages are also interleaved, on which the user may make his own personal notes. Altogether, the book is of such a useful and time-saving nature that no busy analyst can afford to be without it.

W. J. S.

CORRESPONDENCE.

ASBÓTH'S PROCESS FOR THE ANALYSIS OF BUTTER.

To the Editor of THE ANALYST.

DEAR SIR,—This, which is recorded on page 213 of your issue for August last, though excellent is not novel, a similar process having been described by West-Knights (ANALYST, vol. v., p. 155), the only difference being the employment of a lime salt instead of a lead one.

The process for estimating oleic acid is substantially that described in THE ANALYST, vol. xiv., p. 61, the only modification being that the lead salts are precipitated in a cold solution, as was proposed by myself in the *Nederl. Tydschr. v. Pharm., etc.*, 1890, p. 26.

Yours, etc.,

L. DE KONINGH.

THE ANALYST.

NOVEMBER, 1897.

PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS.

DERBY MEETING.

A COUNTRY meeting of the Society was held at Derby on Friday, October 8.

In the morning a visit was paid to the locomotive works and also to the carriage works of the Midland Railway Company, as well as to the excellently-appointed laboratory of Mr. Archbutt, the chemist to the company. Before proceeding to inspect the works, the members had a cordial reception from Mr. S. W. Johnson, the locomotive superintendent of the Midland Railway, who spoke in appreciative terms of the services rendered to railway engineers by the chemical profession.

After lunch, visits were paid to the printing and lithographic works of Messrs. Bemrose and Sons, and to the works of the Crown Derby Porcelain Company.

In the evening the members dined together at the Midland Railway Hotel. Amongst the visitors present were Professor Clowes, president of the Society of Chemical Industry, Mr. James, of the Midland Railway Company, and Dr. Iliffe, medical officer of health to the Corporation of Derby. The president of the Society (Dr. Dyer) referred to the disappointment experienced by all public analysts at the meagre and unsatisfactory nature of the Adulteration Bill introduced to Parliament towards the close of last session by the president and secretary of the Local Government Board, and expressed the hope that every public analyst would impress upon his County Council or local authority the shortcomings of that Bill, in order that, in the interests of the public, such authorities might take steps to endeavour to induce the Government to introduce a better measure, which should really embody, instead of overlooking, the chief recommendations of the Select Committee. Cordial thanks were expressed to Mr. Archbutt and to Mr. White (public analyst for the county of Derby) for the trouble they had taken in organizing a pleasant and successful gathering.

ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

FOOD AND DRUGS ANALYSIS.

The Detection of Formalin in Milk by Means of Phloroglucinol. M. Jorissen. (*Service de Surveillance des Aliments en Belgique*; through *Bull. Soc. Chim.*, 1897, 167.)—Several drops of a 10 per cent. aqueous solution of phloroglucinol are added to about 10 c.c. of the milk, the mixture shaken, and a few drops of a solution of caustic soda or potash added. A red coloration is produced with milk containing as little as 1 part of formalin in 20,000, whilst normal milk gives no reaction.

C. A. M.

Proportion of Mineral Matters and Earthy Phosphates in Cows' Milk. L. Vaudin. (*Ann. de Chim. Analyt.*, ii. [18], 344-347.)—From analyses of the milk from several different breeds of cows fed on a variety of fodder, the author concludes that in normal milk the percentage of mineral matter (ash) ranges within the limits of 7 and 8 grammes per litre, of which 3·3 to 4 grammes consist of earthy phosphates (lime, magnesia and iron), precipitable by ammonia. This is independent of breed, soil, daily yield, etc., but the individuality of the animal and the diet exercise a slight influence on the proportions between the above limits. On the other hand, in milk rendered abnormal by pathological or other influences, the percentage of ash and proteid bodies is increased, but in an irregular manner. For example, in the case of a pregnant cow the milk contained 52·16 per cent. of albuminoids and 8·6 per cent. of ash, and that from one poisoned by oil-cake infested with aspergillus, 44·0 and 8·5 per cent. respectively, whereas the albuminoids in two instances of normal milk amounted to only 36·3 and 41·6 per cent.

C. S.

Determination of Boric Acid in Meat Products. C. Fresenius and G. Popp. (*Zeit. für öffent. Chemie*, iii., 188.)—10 grammes of chopped meat or sausage-meat are rubbed up in a mortar with four to eight times the quantity of calcined sodium sulphate. The mortar with its contents is then placed in a water-bath for about an hour, and, as soon as the mass is dry, some more sodium sulphate is added and the whole ground to a fine powder. This is then digested in 100 c.c. of cold methyl alcohol for twelve hours, with frequent shaking, after which the alcohol is distilled off. Though, as a rule, the boric acid passes over completely in one distillation, it is recommended to extract the residue a second time, using 50 c.c. methyl alcohol. The distillate is made up to 150 c.c. with methyl alcohol, and the boric acid determined in 50 c.c. by titration as follows: The 50 c.c. are mixed with 75 c.c. of water, and 25 c.c. of pure glycerin. The whole is well mixed, and then titrated with $\frac{N}{20}$ soda solution (free from carbonic acid) using phenol-phthalein as indicator. A pale rose colour denotes the end of the reaction. When this appears, more glycerin solution is added, and if the colour does not stand, the titration is continued

further. The number of c.c. of soda used multiplied by 0·0031 gives the boric acid (H_3BO_3) in the titrated quantity of distillate.

Should the sample contain borates as well as boric acid, they would remain in the residue left from the distillation. As most borates (and especially sodium borate) are soluble in methyl alcohol, they can be extracted by this means from the mass, and determined in the evaporated residue, after incineration, by the method of Hönig and Spitz.

The following results show the accuracy of the method:

0·4038	gramme	boric acid	in 10	grammes	chopped meat	yielded 0·4093 gramme.
0·4034	,,	,,	,,	,,	,,	0·4030 ,,
0·4212	,,	,,	,,	,,	,,	0·4264 ,,
0·1600	,,	,,	,,	,,	,,	0·1674 ,,
0·0503	,,	,,	,,	,,	,,	0·0511 ,,

H. H. B. S.

On Wine-Glycerin. R. Kayser. (*Zeit. für öffent. Chemie*, iii., 190.)—As is well known, the glycerin extracted from wines is always in a very impure state. The author examined the glycerin extracted from a white wine by the process given in the memorandum of the German Imperial Chancellor, of June 25, 1896, and found that it contained in round numbers about 20 per cent. of foreign substance.

H. H. B. S.

New Method of Estimating Tartaric Acid in Wine. L. Briand. (*Ann. de Chim. Analyt.*, ii. [17], 321.)—The author proposes to add to the wine (50 c.c.) an excess of pure potassium sulphate crystals, shaking up briskly, and leaving for two days in a cool place. After filtering, the residue is carefully washed with dilute alcohol (15 per cent.), saturated with both potassium sulphate and bitartrate. The correction to be added for the solubility of the bitartrate in the wine after the above treatment is 0·35 gramme per litre. By this means the free tartaric acid present is obtained as bitartrate in addition to that existing in a ready-formed condition in the wine. The results are very concordant, and the other constituents of the liquid do not exert any appreciable influence on the solubility of the precipitate. C. S.

Estimation of Potassium Bitartrate in Wines. H. Jay. (*Ann. de Chim. Analyt.*, ii. [14], 264-267.)—From investigations performed on wines and several other alcoholic liquids containing potassium bitartrate, to compare the accuracy of the Berthelot and Fleurieu (direct precipitation) and Reboul (evaporation) methods, the author concludes that the higher results furnished by the latter are due to a disturbance of the natural equilibrium of the various acids and salts in the liquids in question. The result of this action is that potassium bimalate crystallizes out along with the bitartrate, and gives results in excess of the truth. The error may be demonstrated by evaporating down to 100 c.c. different volumes of liquid containing bitartrate, the results becoming excessive when the reduction in volume is greater than 5 : 1. Consequently he considers the direct precipitation method to be, though inexact, nearer and more regularly approximate to the truth than that of Reboul. The correction found necessary to make good the deficiency in the results of the first-

named method was determined, from liquids containing 1·35 to 2·72 grammes of bitartrate, to be between 0·19 and 0·22 gramme. C. S.

Estimation of Potassium Bitartrate and Free Tartaric Acid in Wines. L. Magnier de la Source. (*Ann. de Chim. Analyt.*, ii. [15], 281-283.)—Referring to the paper by H. Jay (see previous abstract), the author remarks that the error due to potassium bimalate in the Reboul and similar methods may also be occasioned by free malic, tartaric and lactic acids, œnolic acids, and acid salts. This will be greater in proportion to the suddenness with which the evaporated wine, etc., is cooled, but is reduced, by his own method (ANALYST, this volume, p. 217) of slow evaporation at the ordinary temperature.

When the presence of free tartaric acid is suspected, sufficient potassium hydrate should be added to neutralize about one-fifth of the total acidity before concentrating the liquid. A check experiment, without addition of potassium hydrate, will show whether an excess of tartaric acid is present. In this approximate test for the free acid, the best washing liquid is dilute alcohol (30 to 35 per cent.), the bitartrate crystals being but very slightly soluble therein; but for the accurate estimation of the total acid when the wine has been partly saturated by potassium hydrate the solution of potassium bitartrate and sulphate (*l.c.*) is preferable. C. S.

Earth Nut-Oil. S. P. Sadtler. (*Amer. Jour. Pharm.*, 1897, lxix., 490-492.)—Owing to the more extended use of earth nut-meal, the expression of the oil is now undertaken on a commercial scale. The author considers that when freed from the free acid present in the raw state it does not tend to become rancid so readily as olive-oil, and that there is no reason why it should not be used in pharmacy where that oil is now used, the products being equally good, whilst the cost is much less. It is stated that the bulk of Castile soap manufactured in Marseilles at the present day is made from African earth nut-oil.

The cold pressed oil is of a pale, yellow colour. About 38 per cent. was obtained from the Virginian earth nuts, and nearly 10 per cent. more by a second hot-pressing. The following table gives the results of the analyses of oil derived from various sources:

	Oil from Virginia Nuts.	Oil from Spanish Nuts.	Oil from African Nuts.	Oil from Puducheri.	Commercial Oil.
Specific gravity at 15° C.	0·917	0·9175	0·911	0·920	0·9209
Saponification value ...	192·53	190·68	194	193·1	192·1
Iodine value	91·75	94·17	85·6	95	98·4
Hehner value	94·87	95·31	—	—	95·86
Reichert-Meissl value ...	0·48	1·60	—	—	—
Free acid as oleic, per cent.	0·55	0·79	0·62	—	6·20
Cold test of the oil ...	+3° C.	+3° C.	+2° C.	—	+10° C.
Maumené test	56·75° C.	—	—	49° C.	45·5° C.
Melting-point of fatty acids	29° C.	34° C.	30° C.	29° C.	28° C.
Solidifying-point ,,	27·5° C.	32·5° C.	29° C.	25° C.	25° C.

C. A. M.

Colorimetric Determination of Aymi and Isobutyl Alcohols. X. Rocques. (*Ann. de Chim. Analyt.*, ii. [12], 221, 222.)—In the case of wine-brandies, and similar liquids wherein amyl alcohol constitutes the major part of the higher alcohols present, it would be more logical to express the results obtained by the author's colorimetric method of estimation (ANALYST, vol xxii., p. 263) as amyl alcohol. To this end he has plotted curves of the data obtained for amyl and isobutyl alcohols under identical conditions, from which it appears that their colorimetric ratio is ordinarily about 6 : 10. In a 50% wine-brandy containing a quantity of higher alcohols expressed by 1 gramme of isobutylic alcohol, according to the aforesaid method, the actual quantity of amyl alcohol present would be over 1·5 gramme. Bearing this in mind, it is unnecessary to alter the type of comparison in use, since that course would lead to a confusion in the results obtained by different workers.

C. S.

On the Determination of the Caramel on Coffee-berries roasted with Sugar. W. Fresenius and L. Grünhut. (*Zeit. anal. Chem.*, 1897, xxxvi., 225-233.)—The different methods used in this determination all consist in treating the entire berries with a solvent for a certain time.

König's Method.—Ten grammes are twice shaken for five minutes with 200 c.c. of boiling water. Each time the solution is decanted and the berries finally washed with 100 c.c. When cool, the solution is made up to 500 c.c. and filtered. An aliquot part of the filtrate is evaporated, the residue dried at 100°, weighed, and the ash determined and deducted.

Neubauer's Method.—Ten grammes of the berries are moistened with ether, and then treated for fifteen minutes with 400 c.c. of boiling water, with frequent shaking. The solution and washings are made up to 500 c.c., filtered, and the caramel determined as in König's method.

Stutzer's Method.—Ten grammes are shaken in a shaking machine for five minutes, with 250 c.c. of cold water. The liquid (containing the berries) is made up to 500 c.c., filtered, and an aliquot part of the filtrate treated as above. This is the method adopted by the Association of Bavarian Chemists.

Hilger's Method.—Ten grammes are shaken for half an hour each time with three successive portions of 100 c.c. of dilute alcohol (equal parts of water and 90 per cent., by volume, alcohol). The united solutions are made up to 500 c.c., filtered, and the residue and ash determined as in the other methods.

The authors point out that it is necessary to decant the liquid from the berries before filtering, since the extra time considerably increases the relative amount of ash in the extract, which is to be attributed to more of the constituents of the berry being extracted.

These methods were tried on four different kinds of coffee which had been roasted, alone and with the addition of sugar in two proportions. The percentage of residue yielded to the solvents when the berries were roasted alone in the normal manner (*i.e.*, with about 18 per cent. loss) was :

I.

Coffee.	Containing Ash.				Ash-free.			
	König.	Neubauer.	Hilger.	Stutzer.	König.	Neubauer.	Hilger.	Stutzer.
Yellow Java	2·45	2·49	0·92	0·77	1·91	1·75	0·71	0·60
Green Java	3·22	2·32	0·87	1·06	2·38	1·65	0·62	0·73
Blue Java	3·47	2·74	1·81	0·46	2·52	1·97	1·39	0·42
Maracaibo	3·77	2·49	0·66	0·46	2·56	1·83	0·60	0·33

Deducting these values from the total amount of residue obtained when the same coffees had been roasted with sugar, the following amounts of caramel were left:

II.

Coffee.	Sugar added. Per cent.	Containing Ash.				Ash-free.			
		König.	Neubauer.	Hilger.	Stutzer.	König.	Neubauer.	Hilger.	Stutzer.
Yellow Java ...	7½	3·64	2·30	2·46	2·07	2·98	2·01	2·33	1·66
	9	3·82	2·34	2·95	1·35	3·33	2·38	2·95	1·18
Green Java ...	7½	0·95	2·43	2·31	0·38	1·07	2·27	2·27	0·58
	9	4·30	4·24	3·65	1·47	4·17	4·08	3·67	1·54
Blue Java ...	7½	1·79	2·57	1·86	1 16	2·00	2·49	1·99	1·09
	9	3·75	4·76	3·72	2·40	3·57	4·19	3·44	1·04
Maracaibo ...	7½	3·47	3·88	3·29	1·28	3·04	3·24	3·01	1·08
	9	3·99	4·93	4·07	1·87	4·07	4·50	3·62	1·70

These results show that the amount of caramel obtained by Stutzer's method is altogether too low if a deduction is made for the substances extracted from the coffee-berry itself. The other methods show an approximate agreement, but when they differ, that of Hilger usually agrees with one or the other. This fact, and also that the amount of ash passing into solution when the berries roasted without sugar are treated by the different methods is considerably less by Hilger's process than in the methods of König and Neubauer, have led the authors to give the preference to the former. They consider that the most accurate course is to make a determination by Hilger's method, and to deduct from the result a mean constant for the substances in the berries themselves which have dissolved with the caramel. The figures thus obtained, together with those from Stutzer's method (without deduction) are shown below.

III.

Coffee.	Per cent. Soluble Matter from the Whole Coffee-beans.			
	Containing Ash.		Ash-free.	
	Hilger, deducting 1·07.	Stutzer, without deduction.	Hilger, deducting 0·83.	Stutzer, without deduction.
Yellow Java, roasted with 7½ per cent. sugar	2·21	2·84	2·21	2·26
,, ,, 9 ,, ,,	2·80	2·12	2·83	1·78
Green Java, roasted with 7½ per cent. sugar...	2·11	1·44	2·06	1·31
,, ,, 9 ,, ,,	3·45	2·53	3·46	2·27
Blue Java, roasted with 7½ per cent. sugar ...	2·60	1·62	2·55	1·51
,, ,, 9 ,, ,,	4·46	2·86	4·00	1·46
Maracaibo, roasted with 7½ per cent. sugar ...	2·88	1·74	2·78	1·41
,, ,, 9 ,, ,, ...	3·66	2·33	3·39	2·03
Carlsbad Mixture I. (sugar unknown) ...	2·49	2·33	2·31	1·77
,, ,, II. ,, ,, ...	2·30	1·55	2·17	1·37
Vienna Mixture I. ,, ,, ...	1·91	1·91	1·88	1·72
,, ,, II. ,, ,, ...	2·43	2·19	2·02	1·71

C. A. M.

The Alterations produced in Coffee on Roasting. A. Hilger and A. Juckenack. (*Forsch. Ber.*, 1897, iv., 119-135.)—The authors have studied the alterations which take place when coffee is roasted in the usual manner, and after the addition of 8 to 9 per cent. of sugar, according to a German patent. The chief results of their experiments, summarized, are :

1. *Caffeine.*—In the ordinary process of roasting, coffee loses about 21 per cent. of its caffeine, and about twice as much when it is roasted with sugar. This is to be explained by the fact that the sugar is added with the object of glazing the coffee, and that for this purpose it is necessary to caramelize it, which requires a much higher temperature than is reached in the usual method of roasting.

2. *Fat.*—The loss of fat in the ordinary roasting is from 9 to 10 per cent. In the glazing process it amounts to about 20 per cent. As a rule, the different constants of the fat showed an increase on roasting, both in the ordinary way and with the addition of sugar.

3. *Organic Matter.*—In the author's experiments, coffee lost in the mean 11·375 per cent. of organic matter on roasting, and about 10 per cent. in the glazing process, allowance being made in the latter case for the amount of caramel produced. The amount of caramel on the glazed coffee was determined by Stutzer's method. Twenty grammes of the glazed beans were shaken in a machine for five minutes with 500 grammes of water at 20° C. The liquid was filtered, 250 c.c. of the filtrate evaporated to dryness in a platinum dish, and the residue dried and weighed. From the weight obtained the weight of ash was deducted. In this way on coffee glazed with 9 per cent. of sugar there was found 1·40 per cent. of caramel, as against 1·25 per cent. in a sample of Java coffee glazed with 8 per cent. of sugar (*cf.* ANALYST, xx., 249), and the preceding abstract. C. A. M.

A Method of Collecting and Analysing the Gases in Damaged Canned Goods. C. A. Doremus. (*Jour. Amer. Chem. Soc.*, 1897, xix., 733-735.)—A hollow, bevelled, steel needle is fixed in the upper arm of an adjustable clamp, with its point passing through the hole of a rubber cork which rests upon the top of the can. The upper part of the needle is connected by means of a capillary tube with a gas burette or nitrometer filled with water or mercury. The can, which is held between the lower arm of the clamp and the rubber cork, is then punctured by turning the lower screw until the needle pierces the top. The rubber cork makes a tight joint round the needle, and the gases escape gently into the eudiometer, where they are measured and analysed in the usual way. From 60 to 80 c.c. of the gas can often be collected.

When there is a putrid odour, carbon dioxide forms the chief constituent of the mixed gases. In other cases hydrogen predominates, there is no offensive smell, and bacteria are absent, whilst there are indications of the corrosion of the inner metallic surface. Hydrogen has also been found without distinctive signs of corrosion. Occasionally the can is discolored, as though traces of sulphuretted hydrogen had been formed, and reactions of the metals may be obtained with the contents of such tins. C. A. M.

On the Amount of Iron in the Ashes of certain Plants. B. Niederstadt. (*Forsch. Ber.*, 1897, iv., 140.)—This paper gives the figures recently obtained by the author in his analysis of various vegetable products used as food. The amount of iron (as Fe_2O_3) contained in twenty-six samples of American apple cuttings varied from 0·009 to 0·046 per cent., but, as these had all been cut with a steel knife, it was possible that some of the iron might have been derived from the knife.

The mean percentage of ash in the specimens of malt examined was 2·5, and the iron oxide ranged from 0·25 to 0·91 per cent. In unroasted coffee-berries of various origin, Liebig found from 0·019 to 0·044 per cent. of iron oxide, whilst the author's analyses of seventeen samples gave as a rule considerably higher results, the raw berries containing from 0·35 to 0·88 per cent., and the roasted berries from 0·07 to 0·65 per cent. C. A. M.

A Method of Drying Sensitive Organic Substances. C. C. Parsons. (*Jour. Amer. Chem. Soc.*, 1897, xix., 388, 389.)—The process consists in expelling the moisture by putting the substance directly into hot mineral oil. The best results were obtained by the author with a perfectly neutral paraffin oil, of specific gravity 0·920, 435° flash test, 500° fire test, and about 550° boiling-point. The object of a high fire test is that the oil shall be free from volatile matter which would escape with the moisture.

The oil is prepared by heating it to about 250° for some time and keeping it in a closed vessel. The substance to be dried is divided into thin small pieces, weighed, and placed with about six times its weight of oil in an evaporating dish. The dish and oil which have been previously heated to 240°, are, after the addition of the substance, maintained at that temperature for a few minutes after effervescence has ceased. The difference between the weight of the dish and oil plus the substance before the

drying, and of the dish and its contents after drying, gives the moisture. Ordinarily the whole operation may be completed in twenty minutes.

Very satisfactory results have been obtained with soap and wood pulp, and since the substance is perfectly protected from the action of the air by being immersed in the oil, it should be possible to use the process in the case of many substances which cannot be dried in the ordinary way. C. A. M.

Application of Hydrogen Peroxide to the Analysis of Food Stuffs. Lebbin. (*Pharm. Zeit.*, xvii., 1897; through *Deutsche Chem. Zeit.*, 1897, 205.)—This reagent possesses in a high degree the property of converting starch into soluble modifications, and can consequently be employed in all those cases where it is necessary to remove starch from other tissues. If so much potato-starch is added to a solution of hydrogen peroxide as would with a similar quantity of water yield a thin paste, the mixture brought to the boil, and a few drops of ammonia added, considerable frothing ensues, and a clear solution results almost instantaneously. If the solution is immediately neutralized and tested with iodine, the blue colour shows that the action has only proceeded as far as soluble starch; if, however, the solution is boiled for some time, it yields with this reagent the brown-red colour indicative of erythrodextrin. Estimation of the starch dissolved in this way does not, so far, appear to have been attended with entirely satisfactory results; but the process is peculiarly applicable to those cases where the starch-free residue has to be submitted to microscopical examination, since the microscopical appearance of the cellulose structures is entirely unaltered. The author has found the method serviceable in differentiating wheat-flour from rye-flour, also in detecting ergot in flour. W. J. S.

A New Method for Isolating Deutero-albumose. Sigmund Fränkel. (*Monatsheft f. Chemie*, 1897, 433.)—In order to obviate the difficulty of removing large quantities of sodium chloride or ammonium sulphate by dialysis, and to avoid the actual loss of substance entailed in the separation, both of which are inherent in the process proposed for this purpose by Kühne and improved by Neumeister, the author proposes to make use of cupric sulphate. This reagent, though it does not cause the slightest turbidity in solutions of pure deutero-albumose, gives, according to Neumeister, a voluminous precipitate with solutions of 1:500, and a turbidity with solutions of 1:1,000 of deutero-albumose contaminated with proto-albumose. Experiments were conducted with Witte's salt-free peptone, with Finzelburg's albumin- and meat-peptone, and also with pepsin- and trypsin-peptones of the author's own preparation.

A dilute solution of cupric sulphate was added to the solution of the peptone, when a tough coherent precipitate formed, leaving a turbidity which generally disappeared after a few hours' standing. Attempts to separate the copper from the solution by hydrogen sulphide or magnesium failed, but success was attained with barium ferrocyanide. A hot saturated solution of this salt was added until a filtered portion of the proteose solution only showed traces of copper. Before absolutely the

whole of the copper was removed, the mixture was acidulated with acetic acid, warmed, filtered, and the filtrate washed. Solution of barium ferrocyanide was then added drop by drop to the warm filtrate as long as a red precipitate formed; then barium acetate to remove sulphuric acid. By practice it was found easy to hit the exact point when all the cupric sulphate was removed. The solution, after being evaporated to a small bulk, was poured into strong alcohol, dehydrated with absolute alcohol, and washed with ether.

Deutero-proteose prepared in this way gives no turbidity or precipitate with sodium chloride alone, but on the addition of acetic acid partial precipitation ensues; ammonium sulphate also yields an abundant precipitate. It gives neither turbidity nor precipitate with potassium ferrocyanide and acetic acid, nor with copper sulphate, and is therefore free from proto-albumose. Experiment showed that the method was suitable for the isolation of the deutero-albumose obtained in trypsin digestions.

W. J. S.

Examination of Mace. P. Soltsien. (*Zeit. für öffent. Chemie*, iii., 253.)—Attention is called to the difference between Bombay and Banda mace as regards the quantity of matter extracted by ether, after removal of the fat by petroleum spirit, and it is suggested that advantage should be taken of this fact, in order to distinguish between the two. The difference is very considerable, the quantity being about ten times as great with Bombay mace as with true mace. The author has never found more than 4·8 per cent. of matter extracted by ether in a pure Banda mace, and proposes that 5·5 per cent. should be fixed as a safe maximum.

The manipulation is carried out as follows: 10 grammes of powdered mace are exhausted by boiling petroleum spirit in an extraction apparatus. On cooling, an oily portion tends to separate at the bottom of the vessel, and this belongs properly to the extractive matter soluble in ether. The petroleum spirit extract is poured off, the separated oily portion in the flask washed with petroleum spirit and dissolved in absolute ether, and then a second extraction is made with boiling ether. In the ether extract there is also a tendency for a portion to separate out. The extract is poured off, the separated matter washed with ether, and the washings added to the extract, which is then filtered, evaporated, and dried in the water-bath, the residue being weighed.

H. H. B. S.

On Clove-Oil. E. Erdmann. (*Jour. prak. Chem.*, 1897, lvi., 143-156.)—Thom's method of valuing clove-oil (*Verhand. der Gesell. deutsch. Naturf.*, 1891, 184) consists in determining the amount of eugenol after converting it into benzoyl eugenol. The author points out that this is based on the assumption that only free eugenol is present, and proves that by previous complete saponification higher results are obtained owing to the presence of aceteugenol, which he finds to be a constituent of the higher-boiling fractions of clove-oil. After the determination of the eugenol by Thom's method, a further estimation should be made in the following manner: 5 grammes of the clove-oil are saponified by heating over a naked flame for three minutes with 11 grammes of soda lye (29 per cent. NaOH). Alcoholic potash must not be used, or the results will be too low on account of the formation of esters.

After saponification 10 c.c. of water are added, and the estimation completed as in Thom's method. In this way the following percentages of eugenol were obtained:

	Thom's Method.	Total Eugenol.
Clove-oil A ...	... 83·9 ...	... 85·68
B ...	... 82·97 / 82·77 ...	... 84·84
C ...	... 80·2 ...	... 81·9

The difference between the results of the first and second estimations corresponds to a percentage of from 2·1 to 2·6 of aceteugenol. Specimens of clove-stalk-oil were found to contain no aceteugenol, and this appears to be a point of difference between the two oils.

Among other substances found in clove-oil were salicylic acid (which was probably present as acetyl-salicylic acid in combination with the eugenol) and furfural. The latter was extracted from the oil, and also identified by the xylidine reaction (*Ber.* xx., 540). The vapour from the heated oil is brought into contact with filter-paper moistened with a mixture of *m*-xylidine and glacial acetic acid, or with aniline acetate. A still more sensitive reaction for furfural is given by a solution of 5 grammes of β-naphthylamine in 20 c.c. of acetic acid, several drops of which are placed on filter-paper, and a drop of the solution to be tested then added. Strong furfural solutions give an intense purple colour. Dilute aqueous solutions (1 : 10,000) show the reaction strongly, and it is still recognisable in solutions of 1 in 100,000. In this way furfural can be detected in cigar smoke. Both clove-oil and clove-stalk-oil give an intense purple colour.

Characteristic colours are given by other aldehydes with the reagent. Citral gives a reddish yellow; salicylic aldehyde and anisaldehyde a yellow; formaldehyde, propylaldehyde and valeric aldehyde, an extremely faint yellow; acetaldehyde and crotonaldehyde a somewhat stronger brownish yellow. A yellow colour is also obtained with eugenol. C. A. M.

TOXICOLOGICAL ANALYSIS.

The Destruction of Organic Matter in Toxicological Work. A. Villiers. (*Bull. Soc. Chim.*, 1897, 678.)—The substance is placed in a flask with a thistle funnel reaching nearly to the bottom, and a delivery tube passing into a vessel containing water, and is mixed with dilute hydrochloric acid (1 : 2 or 3). Several drops of a solution of a manganese salt and a little nitric acid are then introduced and the flask gently warmed. The gaseous products, consisting of carbon dioxide and nearly pure nitrogen, pass off very regularly, and there is no liberation of malodorous vapours. The operation is completed as in the ordinary chlorate method. Any residue left consists only of fatty matter which has been able to resist the oxidizing action. It is claimed that the method is much more easily carried out than oxidation by means of potassium chlorate and hydrochloric acid.

C. A. M.

ORGANIC ANALYSIS.

Examination of Bees-wax. **R. Henriques.** (*Zeit. für öffent. Chemie*, iii., 274.)—The author has examined Weinwurm's qualitative method for testing the purity of bees-wax (*cf.* ANALYST, this vol., 242). He reports favourably of the process, but proposes to simplify it by a modification, which was suggested by Leffmann and Beam's proposal for the saponification of butter by alkaline glycerin solution (*cf.* ANALYST, 1891, 153; 1892, 65).

The modified process is conducted as follows: A piece of wax about the size of a pea is boiled in a test-tube for three or four minutes with 5 c.c. of alkaline glycerin solution (25 c.c. soda solution of a specific gravity of 1·383 and 125 c.c. pure glycerin). The solution, which is at first quite clear, becomes gradually cloudy. After boiling for about the time mentioned, the oil collects to a layer, and the underlying fluid becomes clear. The bubbles of the boiling mass also now become smaller, and the glycerin commences to distil. As soon as this point is reached, the heating is discontinued. The fluid is now poured into another test-tube, in order to separate it from the unsaponified portion; an equal volume of hot water is added, and the fluid boiled and allowed to cool. In the case of pure wax, the solution will be now either quite clear and transparent, or, at any rate, sufficiently translucent to allow of large printed matter being read through it, as described by Weinwurm. Should, however, on the contrary, as much as 5 per cent. of foreign hydrocarbons be present, the fluid will be quite opaque. With an admixture of only 3 per cent. of ceresin or paraffin, the indication is uncertain, and the further procedure recommended by Weinwurm to meet such cases should be followed. H. H. B. S.

Determination of Paraffin in the Distillates from Lignite, Tar, and Crude Petroleum. **D. Holde.** (*Zeit. angew. Chem.*, 1897, 116, 117.)—The author has confirmed Aisinman's statement as to the comparative insolubility in alcohol of the distillates of high boiling-point from crude petroleum. Thus, from 1 to 1½ litres of alcohol were required to dissolve from 10 to 20 grammes of different Russian oils, whilst at the same time a considerable amount of paraffin also dissolved, and the separated paraffin required a great deal of washing to free it from the adhering oil. It was found that a litre of absolute alcohol dissolved 0·3 gramme of paraffin at 0° C.

The method proposed by Grotowsky, and modified by Engler and Böhm, was found to give the most satisfactory results. The oil is dissolved in as little ether as possible, and the paraffin precipitated with alcohol at a temperature of −18° to −20° C., and washed on a double filter with as little as possible of a mixture of ether and alcohol. During the washing the same low temperature is maintained, and the work is done as rapidly as possible in order to prevent the paraffin dissolving. From one to two hours is required for a complete estimation. Test analyses of mixtures of oil and paraffin gave results in close accordance with theory, and duplicate analyses of the distillates of various lubricating-oil distillates agreed well with each other. C. A. M.

The Normal Urine. **C. Platt.** (*Jour. Amer. Chem. Soc.*, 1897, xix., 382, 383.)—For several years the author has kept the records of all urine analyses, with due regard to the age, sex, and health of the individuals, and his figures, in all cases compared, and in some averaged, with those of foreign observers (of whom fifty-two are named), are given in the table below.

The normal urine is of a pale amber or straw colour. It is clear or has a faint cloud of mucus, and has an "aromatic" odour. The reaction is acid, the acidity in twenty-four hours being equivalent to 2 to 4 grammes of oxalic acid. The specific gravity at 15° C. ranges for adults from 1·015 to 1·025, the average for man being 1·020, and for woman 1·018. The quantity in twenty-four hours varies from 1,100 to 1,600 c.c., with an average for man of 1,450 c.c. (22 c.c. per kilo of body weight), and for woman of 1,250 c.c.

		Averages for Adults.		
		Man.		Woman.
	Normal Urine. Grammes.	Grammes Urine in 24 hours.	Grammes per kilo. of body weight.	Grammes Urine in 24 hours.
Total solids	45·0–65·0	60·0	0·91	51·0
Urea	20·0–50·0	34·0	0·51	30·0
Uric acid	0·3–0·8	0·6	0·009	0·5
Creatinine	0·4–1·3	0·9	0·014	0·8
Hippuric acid	0·4–1·0	0·7	0·010	0·6
Oxalic acid	0·020–0·030	0·025	—	—
Xanthine, sarcine, etc.	0·001–0·010	0·005	—	—
Glycero-phosphoric acid	0·010–0·020	0·015	—	—
Propionic, valeric, caproic, and butyric acids	0·008–0·080	0·040	—	—
Phenol, cresol, etc.	0·005–0·020	0·010	—	—
Sulphur dioxide in ethereal sulphates	0·090–0·500	0·250	—	—
Indoxyl sulphuric acid (calculated as indigo)	0·005–0·019	0·008	—	—
Thiocyanic acid	0·001–0·008	0·004	—	—
Para-oxyphenylacetic, para-oxyphenylpropionic, dioxyphenyl lactic and para-oxyphenyl-glycocholic acids	0·010–0·030	0·020	—	—
Bile salts	0·0–0·010	0·008	—	—
Urobilin, urochrome, etc.	0·080–0·140	0·125	—	—
Carbohydrates (reducing power of normal urine equivalent to an average of 0·3 per cent. glucose)	0·014–0·075	0·044	—	—
Chlorine	5·0–10·0	7·3	0·110	6·0
Phosphorus pentoxide	2·0–3·5	3·0	0·045	2·5
Sulphur trioxide	1·5–3·0	2·2	0·033	1·9
Potassium oxide	2·5–3·5	3·0	0·045	2·8
Sodium oxide	4·0–6·0	4·5	0·068	4·0
Ammonia	0·5–0·8	0·72	0·010	0·6
Calcium oxide	0·2–0·4	0·30	0·0045	0·28
Magnesium oxide	0·3–0·5	0·40	0·0066	0·35
Iron	0·001–0·010	0·007	—	—

There are also traces of the following: Sarcolactic, succinic, glycuronic, and

oxaluric acids; acetone, inosite, cystin, taurin, urorubinogen, urorubin, Giacosa's pigment, scatoxyl-sulphuric acid (often in considerable amount), scatoxyl-glycuronic acid; nephrozymase, pepsin, and other ferments; pseudoxanthine, paraxanthine, heteroxanthine, guanine, adenine, etc.; pyrocatechin, hydroquinone, protocatechuic acid, etc.; silicic acid, carbonic acid, hydrogen peroxide, nitrates, nitrites; and metals —*e.g.*, manganese and copper.

Gases in Normal Urine.

	In 100 Volumes of Gas.	In 1 litre of Urine.
	c.c.	c.c.
Carbon dioxide ...	65·40	15·957
Oxygen	2·74	0·658
Nitrogen	31·86	7·775
	100·00	24·390

C. A. M.

Testing Commercial Albumin. P. Carles. (*Ann. de Chim. Analyt.*, ii. [13], 241).—To examine commercial albumin for impurities, 2 grammes are gradually thinned down with water, and finally made up to 200 c.c., the solution being clear in the absence of coagulum due to over-heating in the manufacture. 100 c.c. are taken and shaken up briskly with 35 c.c. of a 1 per cent. solution of pure tannin and 0·20 gramme of powdered potassium bi-tartrate. Ten to 15 grammes of the turbid liquid are passed through a filter, and the clear filtrate divided equally, a few drops of a 0·5 per cent. solution of pure (Grénet's) gelatin (10 c.c. corresponding to 0·10 of a commercially pure tannin) being added to one portion, and a similar quantity of 1 per cent. tannin to the other.

If both solutions remain clear, then the sample is free from extraneous or coagulated matter. If, however, the gelatin solution produces a precipitate, it is evident that the tannin solution was in excess, and that therefore the albumin is poor and adulterated, the amount of adulteration being indicated by the volume of gelatin solution required to precipitate the tannin. On the other hand, a turbidity in the second portion of the filtrate indicates the presence of a substance (*i.e.*, gelatin), precipitable by tannin, and having a higher coefficient of saturation by that reagent than is possessed by albumin.

The second 100 c.c. can then be freed from albumin by boiling, and any gelatin present thrown down by tannin. Any gums or dextrins employed as adulterants can be separated from the residual liquid by strong alcohol after concentration, and can be estimated in the ordinary manner. C. S.

INORGANIC ANALYSIS.

The Volumetric Determination of Lead. J. H. Wainwright. (*Jour. Amer. Chem. Soc.*, 1897, xix., 389-393.)—This is a modification of the process described in Crookes' "Select Methods," which the author found the most satisfactory of various volumetric methods tried, although it was necessary to alter some of the details before he could obtain concordant results. From 1 to 1·5 grammes of the ore are

dissolved in 10 to 15 c.c. of nitric acid (specific gravity 1·20), the solution neutralized with ammonia in excess, and a considerable excess of acetic acid added. It is then boiled, and potassium bichromate solution in sufficient quantity to precipitate nearly all the lead is run in from a burette. The liquid is boiled until the precipitate becomes orange coloured, after which the titration is finished, the final point being indicated by means of silver nitrate as an outside indicator. The bichromate solution should be standardized on the pure metal, or by means of pure white lead in which the metal has been determined gravimetrically. One c.c. should equal about 0·01 gramme of lead.

The precautions to be observed are:

The solution of the lead salt must be as concentrated as possible, and decidedly acid with acetic acid. There must be absence of other metals, especially such as can exist in lower forms of oxidation. Antimony and tin, unless thoroughly oxidized, and bismuth are particularly to be avoided. During titration the solution should be kept as near the boiling-point as possible. The strength of the bichromate solution should not vary much from that given above, and the test solution of silver nitrate should be dilute, not over 2 to 3 per cent.

The method is specially suitable for such substances as white lead, red lead, litharge, etc. Red lead is dissolved by treating it with nitric acid, and adding a dilute solution of oxalic acid drop by drop until the lead oxide is completely dissolved. If organic matter is present the solution should be filtered before titration. White lead can be dissolved directly in acetic acid, and the solution titrated without filtration. In the case of white lead ground in oil, the sample should be dissolved in dilute nitric acid, the solution boiled, filtered, neutralized with ammonia in excess, and an excess of acetic acid added. The method can also be used with lead bullion, and alloys containing tin and antimony, but the sample must be thoroughly oxidized by repeated evaporation with fuming nitric acid, and the solution filtered before titration.

The results tabulated by the author are concordant, and in close agreement with those required by theory. C. A. M.

The Electrolytic Determination of Cadmium. S. Avery and B. Dales. (*Jour. Amer. Chem. Soc.*, 1897, xix., 379-382.)—Precipitation from a cyanide solution was found the most satisfactory when more than 0·1 gramme of the metal was present (solution 150-200 c.c., and area of electrode for plating 100-150 square cm.), but a strong current was necessary, and the last traces of metal were hard to precipitate. A solution containing 0·0452 gramme of cadmium in the cyanide solution was placed in a current from a thermopile for twenty-four hours. The thermopile had a tension of 4 volts, and an ampèrage of 2, with no resistance other than the ampèremeter. The quantity of cadmium deposited was 0·0446 gramme.

The results obtained by the double oxalate method and by precipitation in acetic acid solution were unsatisfactory, but good results were obtained from a formate solution when certain conditions were rigidly maintained. Not more than 0·1 gramme of cadmium in 150 c.c. of solution could be determined on a surface not less than 100 square cm. The voltage might not exceed 3·4 and the ampèrage 0·15-0·20.

The following is given as a representative determination: To a solution of cadmium sulphate 6 c.c. of formic acid (specific gravity 1·20) were added, followed by potassium carbonate until there was a slight permanent precipitate. This was just dissolved in formic acid, and then 1 c.c. excess of the latter added. The solution was diluted to 150 c.c., put in a weighed platinum dish, and placed in the thermopile current for twelve hours. Highest voltage, 3·3; ampèrage, 0·195.

Weight of Cadmium taken.	Weight of Cadmium found.
0·0454	0·0452
0·0454	0·0454
0·0642	0·0642
0·0642	0·0642
0·0642	0·0641

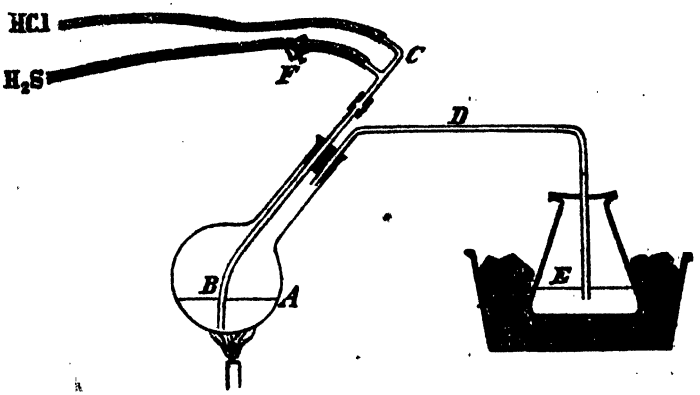
The plates deposited showed no tendency to oxidize, and could be kept in the desiccator for several days. The determinations made in more concentrated solutions, and with stronger currents, often failed to give a good adherent plate. C., A. M.

Separation of Tellurium and Antimony. W. Muthmann and E. Schröder. (*Zeit. anorgan. Chem.*, 1897, xiv., 432.)—As most of the natural compounds of tellurium, including the Hungarian ores Sylvanite and Nagyagite, contain antimony, it is frequently necessary to effect a separation between these two elements; but neither Rose's process—in which the tellurium is precipitated with sulphurous acid—nor Wöhler's—which depends on the formation and subsequent oxidation of potassium telluride—is really trustworthy.

The authors find that tellurium sulphide is an extremely unstable body, and that the metal which results from its decomposition is insoluble in hydrochloric acid not exceeding 20 per cent. in strength; and on these facts they have based the following process. Tartaric acid is added to the hydrochloric acid containing the two oxides Sb_2O_3 and TeO_2 in solution, and a current of sulphuretted hydrogen is passed through it for several hours. The precipitate is warmed and digested with a small excess of potassium sulphide till all is dissolved; and the liquid is then poured into hot 20 per cent. HCl, also containing some tartaric acid, and allowed to rest until the precipitate has settled. The latter is filtered off, washed twice on the filter with hot hydrochloric acid, and afterwards with a weak solution of tartaric acid till it is free from antimony. In order to determine the tellurium, the precipitates must be dissolved in fuming nitric acid, and the metal either weighed as such or as dioxide.

Some examples of the process as applied to known mixtures are given in the paper, with results showing an average deficit of 0·43 per cent. in the case of the tellurium, and of only 0·21 per cent. in that of the antimony; and the authors have also analysed an almost perfectly clean specimen of nagyagite (tellurium glance) from Nagyag, finding its composition to be accurately represented by the empirical formula $Pb_{10}Au_2Sb_2Te_6S_{15}$, which may perhaps be expressed more rationally as $Sb_2Te_3.Au_2Te_3.5Pb_2S_3$. F. H. L.

Quantitative Separation of Arsenic and Antimony. **Oscar Piloty and Alfred Stock.** (*Berichte*, 1897, p. 1649.)—The method is based on the fact that arsenic, when present in a strong hydrochloric acid solution, is readily volatilized by hydrogen sulphide. This fact may also serve to explain the many contradictions found in literature with reference to the precipitation of arsenic as sulphide. If strong hydrochloric acid, containing arsenic trisulphide, is kept in a state of ebullition the arsenic for the most part volatilizes; and in such a solution of either arsenic trioxide or pentoxide no precipitation of sulphide occurs on passing hydrogen sulphide through it; but if the solution is kept boiling, and a current of hydrochloric acid gas is simultaneously passed into it, the arsenic passes over probably as trichloride. From the above it is easily seen that complete precipitation of arsenic by hydrogen sulphide can only take place in a hot solution when this is slightly acid, or in a cold solution when it is strongly acid (*cf.* Bunsen, *Ann. d. Chemie*, 1892, p. 305). In the method proposed the mixture of arsenic and antimony is introduced into a 300 c.c. round-bottomed flask, along with 100 c.c. of strong hydrochloric acid. This flask A is closed with a cork having two perforations, and bearing the arrangement of tubes shown in the accompanying figure, the upper tube serving for the introduction of a current of hydrochloric acid gas, the lower for a current of hydrogen sulphide. A wide-mouthed Erlenmeyer flask E of 600 c.c. capacity, placed in ice, and containing 250 c.c. of dilute hydrochloric acid, serves as the receiver. This flask is covered with a perforated watch-glass, through which the tube D passes and dips under the surface of the liquid. The contents of flask A are brought to a state of lively ebullition, and a rapid current of hydrochloric acid gas is passed through its contents. As soon as the flask has attained the temperature of boiling water, hydrogen sulphide is admitted at the rate of two small bubbles per second. The distillation is rapidly pushed on until only a few c.c. are left in the flask; this occupies from half to three-quarters of an hour. It is best to shut off the current of hydrogen sulphide a short time before the distillation is finished, so that the distilling-flask may be freed from this gas. The whole of the arsenic, which is now present in the receiver as sulphide, is brought on to a filter-paper supported on a perforated filter-plate, dissolved, as also are the portions adhering to the sides of the receiver, in potassium hydroxide solution, and made up to 500 c.c. It is then oxidized with bromine-water, after being slightly acidified with hydrochloric acid, boiled to remove bromine, kept at a temperature of 70° C. for three hours, with a current of hydrogen sulphide passing,



this being maintained until the liquid is cold. After standing for twelve hours, the granular precipitate of arsenic pentasulphide is collected in a Gooch crucible, washed with water, absolute alcohol, carbon disulphide, water-free ether, and dried to constant weight at 105° C.

The contents of flask A, which contains the antimony, are precipitated with hydrogen sulphide, and the precipitate treated in a similar manner to that from the arsenic.

The results obtained are exact. W. J. S.

Qualitative Separations of the Bases of the Second Group in the Absence of Phosphates, etc., by Sodium Nitrite. G. Wynkoop. (*Jour. Amer. Chem. Soc.*, 1897, xix., 434-436.)—Iron, aluminium, and chromium are completely precipitated by sodium nitrite if the solution is boiled for a short time, whilst cobalt, nickel, zinc and manganese are unaffected if a small amount of acid be present in the solution. When iron and chromium are precipitated in the presence of zinc, not more than inappreciable quantities of the latter are simultaneously carried down. The separation of iron from manganese was found to be more accurate and rapid than with barium carbonate. In the cold, iron and some of the cobalt (the latter as double nitrite of cobalt and sodium) are precipitated, while chromium and aluminium do not come down until the solution is heated nearly to the boiling-point. Iron is best precipitated in the ferrous state, since the precipitate is less bulky and gelatinous than the ferric iron, and consequently more easily washed. C. A. M.

A Modification of the Permanganate Method for the Determination of Iron. H. P. Cady and A. P. Ruediger. (*Jour. Amer. Chem. Soc.*, 1897, xix., 575-581.)—Since silver nitrate gives no precipitate in a dilute solution of a chloride in the presence of an excess of mercuric sulphate, and also since silver chloride is moderately soluble in a solution of mercuric sulphate, the authors made experiments to determine whether iron could not be titrated with permanganate in the presence of hydrochloric acid if an excess of mercuric sulphate were added to the solution. The results were very satisfactory, and two methods were worked out:

Method I.—The substance is dissolved in water containing hydrochloric acid, diluted to 100 c.c., boiled and reduced with stannous chloride solution (5 per cent.) added little by little until the solution becomes colorless. Any excess of stannous chloride is oxidized by adding a solution of permanganate drop by drop until a drop of the iron solution gives a faint colour with potassium thiocyanate. One or two drops (not more) of stannous chloride are then added. This whole process should be carried out at boiling heat. The solution is then cooled, 50 c.c. of dilute sulphuric acid added, followed by 35 c.c. of mercuric sulphate solution for every 10 c.c. of hydrochloric acid that was used. This solution is prepared by adding to 200 grammes of mercuric sulphate 80 c.c. of sulphuric acid (conc.), and adding to the paste 800 c.c. of water. To this is added 100 grammes of ortho-phosphoric acid dissolved in a little water, and the whole is diluted to 1 litre. After the addition of the mercuric sulphate,

the solution is diluted to 400 c.c., and titrated with potassium permanganate as usual.

Method II.—The substance is dissolved in 15 c.c. of hydrochloric acid (specific gravity 1·10), 2 c.c. of the mercuric sulphate solution added, the whole heated to boiling, and stannous chloride added in small quantities at a time, until the addition of a single drop produces a turbidity which does not redissolve on boiling. The contents of the flask are rinsed into a beaker, diluted to about 300 c.c., 50 c.c. of dilute sulphuric acid added, and then 45 c.c. of the mercuric sulphate solution; the solution may then be titrated without further cooling.

Method I. can be used in all cases, while Method II. (which is much shorter) can be used when there is not too much insoluble residue which might obscure the reaction at the end of the reduction. The test analyses of ammonium ferric sulphate gave results closely corresponding with those required by theory, but those obtained by Method II. were about 0·1 per cent. too high.

Application of the Process to Iron Ores.—The material is boiled for some time with 10 to 15 c.c. of hydrochloric acid (specific gravity 1·10), with the addition of stannous chloride solution to hasten the decomposition. If Method I. is to be followed, the solution is diluted to 100 c.c. and treated as described. When Method II. is followed, the solution is not diluted, but the 2 c.c. of mercuric sulphate are added, and the process continued as before. If long boiling was required to dissolve the ore, much of the acid may have been driven off, and the precipitate formed on the addition of stannous chloride will not readily redissolve; in such cases a few more c.c. of hydrochloric acid should be added. Ores insoluble in hydrochloric acid may be dissolved after fusion with potassium hydrogen sulphate, and then reduced by either method.

The special precautions to be taken in the process are:

1. The stannous chloride must be in good condition. It should give no precipitate when diluted with twenty volumes of water and boiled.

2. The smallest possible excess of stannous chloride should be present after the reduction is completed.

3. The proportion between the mercuric sulphate and the hydrochloric acid must be carefully adjusted.

It is stated that, in the case of soluble salts, less than three minutes has been found sufficient time for solution, reduction, and titration. C. A. M.

The Employment of Copper Salts in the Estimation of Certain Elements in Cast Iron and Steel. A. Carnot and E. Goutal. (*Ann. de Chim. Analyt.*, ii. [16], 301-305.)

1. *Carbon.*—The rapidity of the ordinary method of attacking the metal by a solution of a cupric salt may be increased by the following modification: From 2 to 5 grammes of the sample, either in lumps or cuttings, are placed in a conical 300 c.c. flask, with a base 12 cm. in diameter, and containing a perforated disc (11 cm.) of platinum to induce electrical action. For each gramme of metal 50 c.c. of a 30 per cent. solution of cupric potassic chloride are taken, 4 or 5 drops of hydrochloric acid

added, and, after boiling for a few seconds, cooled down to 60° C. The metal is then introduced, and the temperature raised to 90°-95° C. To keep the liquid agitated, and at the same time to prevent oxidation of the iron, a current of carbon di-oxide is passed through it continuously. Under these conditions the attack is rapid, fine clippings being dissolved in about twenty minutes, and 5 gramme lumps in an hour and a half. When the precipitated copper is dissolved, the residual carbon is brought by means of the solvent on to an asbestos filter, washed first with acid cupric chloride solution, then with hot water, and burned without previous desiccation. The results are concordant with those furnished by the longer method.

2. *Sulphur.*—The same procedure is adopted, but the washed precipitate is treated with a mixture of 5 c.c. of hydrochloric acid, 15 c.c. of water, and 1 c.c. of bromine in the cold, the temperature being after some time raised slowly to boiling to expel the bromine. After neutralizing the filtered liquid with ammonia and adding a few drops of hydrochloric acid, the sulphur is precipitated as barium sulphate, and either weighed direct, or first purified from slight traces of silica by the addition of hydrofluoric acid and evaporation with a few drops of sulphuric acid.

3. *Phosphorus.*—The metal is attacked by a *neutral* solution of cupric-potassic chloride in the same apparatus, and the residue, after washing, is treated with nitric acid and bromine; then filtered and boiled with a few decigrammes of chromic acid to destroy the carbides. After neutralizing with ammonia, a few drops of nitric acid are added, followed by 50 c.c. of nitro-molybdic reagent at about 45° C., to precipitate the phosphoric acid. Experience has shown that when the temperature is very moderate, and the amount of iron present very small, the precipitate is normal, and may be washed, dried at 100° C., and weighed direct. The silica is eliminated, and there is no necessity for evaporation to dryness; and any arsenic present is dissolved by the copper salt.

4. *Chromium.*—When the solvent is perfectly neutral the chromium is left undissolved, and may then be determined from the washed and calcined residue by acting on the latter with sodium peroxide, taking up with water, boiling and estimating the chromic acid. The method, though advantageous in the case of steel with a low content of chromium, is less so when the metal is poor in carbon, the chromium in such cases being partly dissolved by the copper salt.

5. *Tungsten and Titanium.*—These are also left undissolved in the residue, in the condition of oxides when in small quantity, but partly in the metallic state when present in larger amounts.

The calcined residue, freed from silica by hydrofluoric acid, is attacked by a mixture of potassium nitrate and carbonate (8 : 2), and taken up with water. The tungstic acid is thereby completely dissolved, and is then thrown down as mercurous tungstate, which is washed, calcined in an oxidizing flame, and finally weighed as pure tungstic acid. The titanic acid remains in the residue, and is determined by fusing with potassium bisulphate, reducing the aqueous solution by sulphur dioxide, and precipitating by boiling with sodium acetate.

When the precipitates are very small, their constituents may be distinguished (after fusion with $KHSO_4$, heating with a few drops of sulphuric acid, and cooling) by their colour reaction with hydroquinone, titanic acid giving a crimson-red and

tungstic acid an amethyst-violet. This reaction enables traces of tungstic acid (too small to be found by any other method) to be detected in the original residue, if it be first treated with concentrated ammonia, evaporated, and the hydroquinone test as given above applied. C. S.

The Impurities of Commercial Calcium Carbide. H. Le Chatelier. (*Bull. Soc. Chim.*, 1897, 793, 794.)—After calcium and carbon, the two most abundant elements in commercial calcium carbide are silicon and iron. The latter is present in the smallest proportion, and is exclusively combined with the silicon. On mixing the carbide with water and treating the insoluble residue with methylene iodide, small crystals of iron silicide, $SiFe_2$, settle out.

The excess of silicon above that required by the iron may be in combination with either the calcium or the carbon. If there is an excess of carbon over calcium, silicide of carbon will be found, crystallized in hexagonal plates, and usually of a blue colour. It will be seen floating on the surface of the methylene iodide, together with the excess of graphite.

If, on the other hand, there is an excess of calcium over carbon, calcium silicide will be found disseminated throughout the mass of the carbide in metallic grains having the colour and appearance of zinc. These can be isolated by treating the carbide rapidly with a large excess of cold water, separating the heavier residue by lixiviation and washing it for several seconds with dilute acetic acid. The final residue consists of iron silicide, and the larger grains of calcium silicide. In this case there can be neither graphite nor silicide of carbon, since there must be an excess of calcium for the formation of calcium silicide.

Apparently there are two distinct silicides of calcium One is not easily attacked by nitric acid, but is readily decomposed by hydrochloric acid, with the formation of a yellow insoluble body—the silicone of Wöhler. The other is readily attacked by nitric and acetic acids, and gives with hydrochloric acid a white compound, which, like silicone, dissolves in potash, with an abundant liberation of hydrogen. The yellow and white compounds have compositions corresponding to the formulæ $Si_2O_4H_4$ and $Si_2O_3H_4$ respectively. C. A. M.

Determination of Lime in Calcined Magnesite. R. Hefelmann. (*Zeit. für öffent. Chemie*, iii., 193.)—Whilst the separation of a small amount of magnesia from a large amount of lime presents no particular difficulty, the reverse operation is not so simple a matter. The following process is recommended as giving satisfactory results, the separation being complete without the need of having to resort to a second precipitation:

1 gramme of magnesite is taken and the silica separated in the usual way. The iron is then removed by double precipitation, the filtrate slightly acidified with hydrochloric acid, and the solution made up to 500 c.c. To 250 c.c. of the solution are added 3 grammes of pure ammonium chloride and ammonia to alkaline reaction. 250 c.c. of water are then added, and the lime precipitated in the solution, while boiling, by 2 grammes of ammonium oxalate dissolved in 100 c.c. of water. When

precipitated in this way, the lime is found to be quite free from both magnesium hydrate and magnesium ammonium oxalate. H. H. B. S.

The Determination of Thorium in Thorite. E. Hintz and W. Weber. (*Zeit. anal. Chem*, 1897, xxxvi., 27-31.)—This method is based on the facts that thorium can be precipitated from slightly acid solution by means of oxalic acid, that thorium oxalate is soluble in ammonium oxalate, and that it can be precipitated from such solution by means of hydrochloric acid or ammonia.

One gramme of the mineral is decomposed with concentrated hydrochloric acid and the liquid evaporated to dryness to separate silica. The residue is moistened with 2 c.c. of concentrated hydrochloric acid and the silica filtered off and washed. Copper or lead, if present, are then removed by precipitation with sulphuretted hydrogen, the filtrate boiled to completely expel the excess of reagent, and made up to 200 c.c. A solution of oxalic acid (containing 1 gramme) is added to the hot solution, and after standing for two days the precipitate is filtered off, washed, and digested for several hours on a boiling water-bath with 60 c.c. of a cold saturated solution of ammonium oxalate. The liquid is diluted to 300 c.c., allowed to stand for two days, then filtered, and the filter washed with water containing a trace of ammonium oxalate. The filtrate is heated and 5 c.c. of concentrated hydrochloric acid (specific gravity 1·17) added.

Any residue remaining after the treatment with ammonium oxalate is again digested with 20 c.c. of the ammonium oxalate solution, the liquid made up to 100 c.c. and 1·7 c.c. of concentrated hydrochloric acid added. Should there be a precipitate, the treatment with ammonium oxalate is repeated a third time, and, if necessary, a fourth time.

The precipitates of thorium oxalate formed by the addition of hydrochloric acid are left for two days, then collected on a filter, washed with water containing a little hydrochloric acid, ignited and weighed.

The thoria, as thus obtained, always contains a certain amount of foreign earths, since the oxalates of cerium and yttrium are not completely insoluble in ammonium oxalate. The weighed precipitate is therefore dissolved by boiling with strong hydrochloric acid; or if the thoria cannot in this way be completely dissolved, it is fused with acid potassium sulphate, the melt dissolved in water containing hydrochloric acid, ammonia added, and the precipitate filtered off, washed, and dissolved in hydrochloric acid.

The hydrochloric acid solution is evaporated to dryness, the residue dissolved in water containing 2 or 3 drops of dilute hydrochloric acid, and the solution (about 300 c.c.) boiled for several minutes with 3 or 4 grammes of sodium thiosulphate. When cold, the precipitate is filtered off and washed. Ammonia is added to the filtrate, and the resulting precipitate separated and dissolved in hydrochloric acid. The solution is evaporated to dryness, the residue taken up in a little water, and the liquid boiled with a hot concentrated solution of ammonium oxalate. After being left for some time in the cold, the precipitate (oxalates of cerium and yttrium) is filtered off, washed, ignited, and weighed. This last weight deducted from the weight of the crude thoria gives the amount of pure thoria.

In exceptional cases of very impure thorite the residue from the ammonium oxalate must be dissolved in hydrochloric acid, or, if necessary, fused with acid potassium sulphate, and the hydrochloric acid solution obtained as described above. This solution is evaporated to dryness, the residue taken up in a few drops of hydrochloric acid, the solution diluted to about 100 c.c., and boiled with sodium thiosulphate. The precipitate is dissolved in hydrochloric acid, the solution evaporated to dryness, the residue taken up in very dilute hydrochloric acid, the thorium precipitated with oxalic acid, and the precipitate united to and weighed with the main precipitate.

Since the precipitation of thorium with sodium thiosulphate is sometimes incomplete, ammonia should be added to the filtrate. The resulting precipitate, consisting of cerium oxides and yttria, with trifling quantities of thoria, is dissolved in hydrochloric acid, the solution evaporated to dryness, the residue taken up in water, the solution boiled, and a hot concentrated solution of ammonium oxalate added. The solution is diluted, allowed to stand for twenty-four hours, the thorium precipitated with ammonia, and the precipitate separated, washed, ignited, and weighed with the main precipitate. C. A. M.

Volumetric Estimation of Iodides with Iodic and Arsenious Acids. F. A. Gooch and C. F. Walker. (*Zeit. anorgan. Chem.*, 1897, xiv., 423.)—This process somewhat resembles Riegler's (ANALYST, xxii., 25), in that the iodide is decomposed by iodic acid; but after the liquid has been made alkaline, the whole of the liberated iodine—five-sixths of which is derived from the iodide itself—is titrated by means of arsenious acid. In order to avoid loss of halogen, the operation is conducted in a modified form of Drexel's wash-bottle, in which the inlet tube carries a funnel and a stopcock, while the outlet is fused to a set of Will and Varrentrapp's nitrogen bulbs. The liquid containing from 0·08 to 0·4 gramme of the iodide to be analysed is measured into the bottle, 5 c.c. of a 5 per cent. solution of potassium iodate added, and the mixture diluted to 150 c.c. The stopper is then inserted in its place, and the bulbs are half filled with 5 per cent. potassium iodide. 5 c.c. of 1 : 3 sulphuric acid are run in through the funnel, the cork closed, and the vessel gently shaken. An excess (10 c.c.) of a saturated solution of potassium bicarbonate is next introduced gradually, the cock closed once more, and, without allowing any of the liquid in the bulbs to run back before neutrality is reached, the flask is cautiously agitated till all violet vapours have disappeared. Finally the bulbs are emptied into the flask, the stopper is removed, and all its parts are rinsed into the main portion of the liquid. This is titrated with decinormal arsenious acid till it becomes colourless, and the exact end-point of the reaction is determined with iodine and starch.

It is important that the volume of the liquid at the time of acidification be exactly 150 c.c., and that there be no appreciable amount of chlorides or bromides present; but in other respects alterations in the proportions of the different reagents seem of little moment. The process is manifestly best adapted for the valuation of approximately pure potassium iodide; in this case the factor 0·01383 at once converts the c.c.'s of arsenious acid into grammes of KI. F. H. L.

Purification of Hydrochloric Acid. E. Gravier. (*Rev. de Chim. Analyt. Appl.*, v. [17], 329-330.)—By distilling the hydrochloric acid as rapidly as possible until some two-thirds has passed over, the author finds the residual portion to be entirely free from arsenic. C. S.

A Colour Reaction for Di-Sulphuric Acid. E. Barral. (*Bull. Soc. Chim.*, 1897, 744, 745.)—When benzene-hexachloride para-dichloride ($C_6Cl_6.Cl_2$) is added little by little in a state of fine division to sulphuric acid containing the di-sulphuric acid, a brilliant reddish-violet colour is produced. On the addition of water or sulphuric acid, or even on exposure to moist air, the colour disappears as the acid becomes transformed into ordinary sulphuric acid. That the coloration is not due to impurities in the Nordhausen acid is proved by the fact that pure SO_3, prepared by passing $SO_2 + O$ over spongy platinum, gives the reaction on exposure to moist air.

This disappearance of the red colour as ordinary sulphuric acid is formed, is used by the author as the basis of a volumetric method for determining di-sulphuric acid. A little powdered C_6Cl_8 is added to the Nordhausen acid, which is shaken until the colour appears.

Standard sulphuric acid (acid of 66° with about one-tenth of its weight of water) is then added drop by drop until the colour disappears, and from the amount required the quantity of di-sulphuric acid in the Nordhausen acid is readily calculated.

This method of direct titration gives good results when the acid is colorless, or nearly so, but with many of the Nordhausen acids of commerce it is not so sensitive.

C. A. M.

Reactions of Sulphoxyarsenic Acids. L. W. McCay. (*Chem. Zeit.*, 1897, xxi., 487.)—If a moderately dilute solution of sodium orthomonosulphoxyarseniate is treated with strontium chloride, a white amorphous precipitate consisting of $NaSr_2(AsO_3S)_2,17Aq$ immediately falls, which becomes crystalline on shaking. Similarly, on adding barium chloride to the disulphoxy salt of sodium, a glittering white precipitate of $Ba_3(AsO_2S_2)_2,7Aq$ is rapidly produced. In mixtures of the two acids the di- salt may be tested for with barium after removal of the mono- acid by agitating with strontium chloride for five minutes as above. Since the sulpharsenites and sulpharseniates of the alkaline earths are all soluble in water, these reactions are not impeded by the presence of large quantities of the former. Calcium chloride behaves analogously to the strontium compound, therefore sodium orthodisulphoxyarseniate may be employed to separate barium from strontium or calcium.

F. H. L.

Estimation of Nitrous Acid and Nitrites. B. Grützner. (*Arch. Pharm.*, 1897, ccxxxv., 241; through *Chem. Zeit. Rep.*, 1897, 172.)—This process depends on the reaction $3HNO_2 + HClO_3 = 3HNO_3 + HCl$. 0·1 or 0·2 gramme of the nitrite is dissolved in 500 cc. of water, treated with about 0·5 gramme of potassium chlorate free from chloride and a definite amount of decinormal silver nitrate, strongly

acidified with nitric acid, well shaken up for some minutes, and the excess of silver titrated with sodium chloride. One c.c. of decinormal silver is equivalent to 0·0142 gramme of HNO_2 or 0·0114 gramme of N_2O_3.

Similarly the N_2O_4 in fuming nitric acid may be determined by diluting 5 c.c. of the sample with 750 c.c. of water, and proceeding as above. Three molecules of N_2O_4 correspond to 1 molecule of $AgNO_3$, or 1 c.c. of decinormal silver equals 0·0276 gramme of nitrogen tetroxide. F. H. L.

An Extremely Sensitive Reaction for Nitrites, and its Quantitative Application. E. Riegler. (*Zeit. anal. Chem.*, 1897, xxxvi., 377, 378.)—In a former communication (*Zeit. anal. Chem.*, xxxv., 677) the author recommended naphthionic acid as a sensitive reagent for nitrous acid. In place of this he now describes a reagent twenty times more sensitive. This is prepared by shaking 2 grammes of chemically pure sodium naphthionate and 1 gramme of β-naphthol (purissim.) with 200 c.c. of water, and filtering. The solution is colorless, and can be preserved in the dark without alteration.

The test is applied by adding to 10 c.c. of the liquid to be examined 10 drops of the reagent and 2 drops of concentrated sulphuric acid, and shaking the mixture several times. On now adding about 20 drops of ammonia, a rose-coloured ring is formed at the junction of the liquids, and on shaking the tube the whole liquid becomes red or rose-coloured, according to the amount of nitrous acid present. The reaction depends on the conversion of the naphthionic acid by means of the nitrous acid into diazo-naphthalin-sulphonic acid, which forms with β-naphthol and ammonia a red diazo colouring matter. One part of nitrous acid in a million can be detected in this way.

In using the reaction for the quantitative determination of nitrites, a standard solution is prepared by dissolving 0·406 gramme of pure dry silver nitrite in hot water, adding sodium chloride, and, after cooling, diluting to a litre. When the precipitate has settled, 10 c.c. of the clear solution are removed with a pipette, and made up to a litre. 100 c.c. of this solution (containing 0·0001 gramme of N_2O_3) are placed in a flask, 20 to 30 drops of the naphthol reagent and 4 drops of concentrated sulphuric acid added, the mixture well shaken, about 20 drops of concentrated ammonia solution added, and the liquid again well shaken. The colour of the resulting liquid is compared by means of a colorimeter with that given under the same conditions by 100 c.c. of the liquid to be examined. If the latter contain more than 0·0001 gramme of N_2O_3 in 100 c.c., it must be previously diluted with distilled water. The standard solution must be freshly prepared before each determination. In this way it is possible to estimate 0·00005 gramme of N_2O_3 in a litre of water.

The naphthol reagent may also be used in the dry state. Equal parts of naphthionic acid and β-naphthol are intimately mixed in a mortar, and a pinch of the powder used instead of the 20 drops of the solution. The powder has the advantage of keeping absolutely without alteration, whilst the reaction given by it is equally delicate. C. A. M.

Preservation of Solutions of Oxalic Acid. **E. Fricke.** (*Chem. Zeit.*, xxi., 26, 243.)—This paper deals with the instability of $\frac{N}{100}$ oxalic acid, which, it is said, can be prevented by the addition of boric acid to the solution.

H. H. B. S.

Note on the Analysis of the Sodium Sulphide used in Leather-dressing. **F. Jean.** (*Ann. de Chim. Analyt.*, ii. [18], 341-343.)—A sample of sodium sulphide that had produced spots (recognised as due to iron) on doe-skin during the cleansing and plumping processes was found to form a green aqueous solution, depositing a sediment of iron sulphide, whilst a second sample containing an equal amount of iron gave a yellow solution, without yielding a deposit of sulphide or spotting the skins. For determining the sodium thiosulphate and the excess of sulphur in these samples, the following method was elaborated. Ten grammes of an average sample of the crystallized substance are dissolved in water, the iron sulphide deposit being determined in the insoluble portion. The filtrate is made up to 1 litre, and 10 c.c. of this are titrated with $\frac{N}{10}$ iodine and starch paste to determine the *total sulphur* present. A second 10 c.c. are mixed with 30 c.c. of water and a quantity of ammonium sulphate solution (6·7 grammes $(NH_4)_2SO_4$ per litre) equal to the volume of iodine used in the first test, and distilled, the retort being connected with the upper end of a vertical condenser, the lower end of which dips into 2 c.c. of $\frac{N}{10}$ acid. The operation is suspended when the distillate coming over ceases to have an alkaline reaction. After boiling, to expel hydrogen sulphide, the excess of acid in the receiver is titrated with $\frac{N}{10}$ alkali and litmus, 1 c.c. of the acid saturated by the distilled ammonia corresponding to 0·0039 gramme of *sodium sulphide*. The residual liquid in the still is cooled and titrated with $\frac{N}{10}$ – iodine, 1 c.c of which reagent = 0·0079 of *sodium thiosulphate*. The difference between this and the former iodine titration is calculated as sodium sulphide. By deducting from this the amount determined in the acid titration, a difference is obtained which represents in terms of Na_2S the excess of sulphur present as *polysulphide*, and may be calculated to sulphur (100 Na_2S = 41S). When the monosulphide is pure, the results of the iodine and ammonia tests will be identical.

The composition of the two samples examined was:

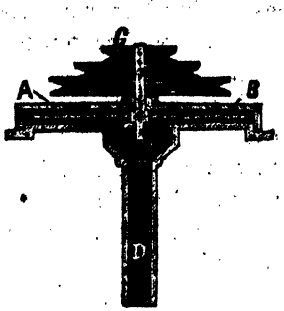
	Green Sulphide.	Yellow Sulphide.
Water	63·24	60·32
Sodium sulphide	31·20	28·08
Sulphur in excess	0·53	1·20
Sodium thiosulphate	4·70	10·00
Iron sulphide	0·33	0·40

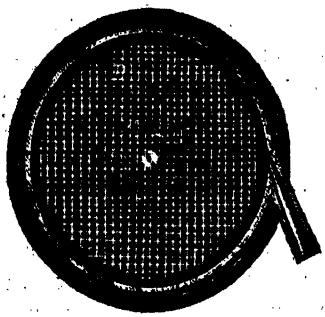
Two hitherto unrecorded observations were made in the course of the investigation, viz., (1) That when a solution of an alkaline monosulphide is boiled with an excess of ammonia, part of the sulphur is volatilized as ammonium sulphide. (2) In *boiling* sodium thiosulphate with excess of ammonium sulphate, ammonium sulphide is liberated and sulphur deposited. This decomposition, however, does not occur in an alkaline medium.

C. S.

APPARATUS.

A New Laboratory Turbine. H. Tryller. (*Berichte*, 1897, p. 1729.)—In this motion is produced by the friction of a jet of water which impinges on a disc of wire gauze, in contradistinction to the usual form where the rotation is caused by the impulse of the jet against a series of floats attached to a wheel. The new form works much more evenly and noiselessly than the older forms, and the rate of motion, as governed by the supply of water, is under better control. The accompanying illustration shows the general form of the apparatus:

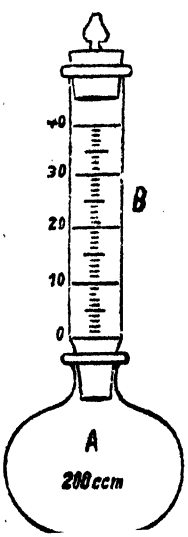




A is the cylindrical case, 100 mm. in diameter and 5 mm. high. The interior is almost completely filled by the disc B, which consists of a double layer of the ordinary iron wire gauze used in the laboratory. This is mounted on an axle, to which the pulley C is attached. The jet of water is directed tangentially towards the periphery of the disc, and is discharged at its centre into the tube D. The apparatus is manufactured by Kähler and Martini, Berlin. W. J. S.

Apparatus for Rapidly Estimating Fatty Acids and the Hehner Number. G. Possetto. (*Boll. Chim. farmac.*, 1897, p. 225; through *Chem. Zeit. Rep.*, 1897, p. 197.)—This consists of a flask of 200 c.c. capacity, with a stoppered tube ground in, as seen in the figure, graduated into forty divisions. The dissolved soap having been decomposed by dilute sulphuric acid in the flask, the tube is fixed in position, the liquid made up to the zero mark on the scale, and ether poured in to the thirty-fifth division. After well shaking and being allowed to settle, an aliquot part of the ethereal solution is removed, evaporated to dryness, and the fatty acids weighed. For ascertaining the Hehner number, the butter or other fat is first saponified in an alcoholic potash solution.

W. J. S.



REVIEWS.

ATLAS AND ESSENTIALS OF BACTERIOLOGY. By K. B. LEHMANN and RUDOLF NEUMANN. (London, Baillière, Tindall and Cox.) Price 12s. 6d.

The first portion of this admirable work consists of a series of sixty-three plates, beautifully executed in colours, which give the appearances exhibited by superficial and deep-seated colonies when grown on gelatin and agar media, as well as in potato cultures; also illustrations of the general appearances in streak and stab cultures, a separate representation being given of the puncture canal and of the surface of the punctured medium in the latter form of culture. These are in many cases supplemented by woodcuts of the organisms. The second portion of the book contains a description of the morphology, vital conditions, sporulation, etc., of bacteria, followed by an account of the effects produced by them, either through the enzymes which they secrete, or simply by metabolism; of the production of pigments, toxins, etc. The technical appendix, which concludes the work, contains a number of useful hints for the bacteriologist when engaged in the actual work of cultivation, but it is evidently assumed that the reader already possesses some rudimentary knowledge of the methods of bacteriological research. These hints are all the more valuable because they bear the impress of being the outcome of long practical experience. The language employed, though occasionally somewhat idiomatic, is intelligible. The work cannot fail to be a valuable addition to the library of the bacteriologist.

W. J. S.

BACTERIOLOGICAL DIAGNOSIS. By ST. GEORGE REID. (London, Baillière, Tindall and Cox.) Price 3s. net.

This is an analytical key for the identification of all the known bacterial micro-organisms, arranged, to use a rough simile, much on the same lines as the tables found in works on qualitative analysis for the identification of the various bases and acids. The tests employed for differentiating the various bacteria are their forms—rods, cocci, etc.—the form of the colonies, their action on the gelatin medium, colour of the colonies, etc. The author is to be commended on the ingenuity displayed in the general arrangement of the key, which seems well adapted for fulfilling its intended purpose. The work will be found useful by those engaged in bacteriological work.

W. J. S.

THE ANALYST.

DECEMBER, 1897.

PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS.

THE monthly meeting of the Society was held on Wednesday evening, November 3, in the Chemical Society's Rooms, Burlington House, the President, Dr. Bernard Dyer, occupying the chair.

The minutes of the previous meeting were read and confirmed.

The following gentlemen were proposed for election as members: Mr. S. H. Collins, F.I.C., Dehra Dun, N.W.P., India, Assistant Agricultural Chemist to the Indian Government; Professor James Hendrick, B.Sc., F.I.C., the University, Aberdeen, District Agricultural Analyst for the County of Aberdeen; Mr. W. H. Jackson, Analyst to the West Yorkshire Coal and Iron Company; and Mr. E. H. Roberts, Analyst to the Dairy Supply Company, Ltd.

Dr. W. J. Russell, F.R.S., was unanimously elected as honorary member of the Society.

The following papers were read:

"The Estimation of Acetates in the Presence of Inorganic Salts." By Bertram Blount.

"The Estimation of Carbonic Acid in Natural Waters." By C. A. Seyler, B.Sc.

"The Detection of Gelatin in Cream;" "A New 'Milk-Preservative';" "A New Milk-Adulterant." By A. W. Stokes.

"Note on the Graduation of Leffmann-Beam Bottles." By G. E. Scott-Smith and A. B. Searle.

"An Improved Milk Scale."

THE DETERMINATION OF ACETIC ACID IN THE PRESENCE OF INORGANIC SALTS.

BY BERTRAM BLOUNT, F.I.C.

(*Read at the Meeting, Nov.* 3, 1897.)

A SHORT time ago I had occasion to determine the quantity of acetic acid present as an acetate in a mixture composed essentially of magnesia, magnesium chloride, and lead acetate, the amount of the last-named ingredient being relatively small. The question appears simple, but, as a matter of fact, required some little consideration and experiment before a satisfactory method was devised.

It may be observed that, acetic acid being a comparatively strong organic acid,

no method such as is commonly used to estimate weak organic acids in the presence of mineral acids (*e.g.*, by titration with selective indicators) was available. The most obvious way, therefore, was to eliminate the chloride as silver chloride, and determine the acetic acid in the filtrate in any convenient manner.

But it was by no means certain that if the mass, which as a whole is sparingly soluble, were extracted with water, it would yield all its acetate. The natural solvent, nitric acid, was not to be thought of, as the separation of this acid from acetic acid is not easy. Hydrochloric acid would require the use of an inconveniently large quantity of silver salt for precipitation. Treatment of the original substance with caustic soda or sodium carbonate, and obtaining all acids as sodium salts, presented some attractions; but, on consideration, it appeared that the bulky mass of magnesia, magnesium hydroxide, and lead hydroxide, which would be left, would be apt to retain soluble salts, including the acetate sought for.

Accordingly, the mixture was dissolved in the cold in sulphuric acid, the chloride precipitated with silver sulphate, and the filtrate distilled into baryta water. The excess of baryta was precipitated by CO_2, and the solution containing the barium acetate evaporated to dryness and the residue weighed. The percentage found fell far short of the truth. It appeared that silver acetate, which is a sparingly soluble salt, could not be separated from the large quantity of silver chloride, unless enormous quantities of washing water were used, making the subsequent distillation tedious and troublesome. Separation by means of silver was, therefore, abandoned.

The plan ultimately adopted was simple and completely satisfactory. It depends on the solubility of sodium acetate in alcohol. The mixture containing magnesia, magnesium chloride, lead acetate, and the products of their interaction, was distilled with excess of sulphuric acid, the distillation being pushed to a point at which the sulphuric acid began to fume. This is necessary in order to ensure driving over the whole of the acetic acid. The distillate was received in a small excess of caustic soda. At the end of the operation the receiver contained sodium chloride, sodium acetate, possibly some sodium sulphate and excess of caustic soda. It was evaporated to a small bulk (but not to dryness), absolute alcohol was added, the whole well stirred, filtered, and washed with absolute alcohol. The alcoholic solution contained sodium acetate and caustic soda. Of these, the latter was eliminated by treatment with CO_2, and on evaporating the filtrate sodium acetate was obtained. The latter still retained a small amount of impurity, and was therefore distilled from a very small retort with excess of sulphuric acid into baryta water, the barium acetate being ultimately separated and weighed.

The method tried on known mixtures proved to be accurate. It appears to be generally applicable, save in the presence of nitric acid. In this case it would probably be best to reduce the nitric acid to ammonia and proceed in the manner already indicated, but I have not had occasion to test the practicability of this plan.

Discussion.

Mr. Allen thought that Mr. Blount had rendered a great service by bringing forward the results of his experience in this matter, and by his sagacity in solving a practical difficulty. It was, unfortunately, not uncommon for the text-books to assume

that questions of this kind were very easy to deal with, whereas they often presented very curious difficulties in practice. While hearing the middle stages of the paper, he had felt rather curious as to how far it would fall into conformity with the lines which he, up to a certain point, would have himself adopted. He did not think the use of alcohol would have occurred to him as necessary. He would have thought it quite possible to acidulate the original material with sulphuric acid, and add sulphate of silver, which would give a precipitate of chloride of silver and sulphate of lead, together with a liquid which could be filtered from the precipitate. There would probably be some mechanical difficulty in the manipulation, but apart from this he would have thought that the acetic acid could be distilled over satisfactorily. He had lately been trying how far certain processes were applicable quantitatively for the separation of some of the commoner metals from the ashes of organic matters, like vegetables, meat, etc. (for instance, for the estimation of tin in tinned meat, zinc in cheese, and so forth). His object was to devise a comparatively simple means of determining, in the same portion of ash, zinc, copper, tin and lead, or any one of the four, and it was astonishing how difficult the problem ultimately proved to be. It would seem at first sight a very simple matter to estimate the lead in a mixture of sodium phosphate, calcium phosphate, lead sulphate and sodium chloride, but, as a matter of fact, a simple process for the estimation of lead and tin in the presence of sulphates, chlorides and phosphates was a thing that required a good deal of thinking and working out. As an instance in point, he might mention that Mr. Hehner was accustomed to estimate tin in tinned meat by boiling the ash with hydrochloric acid. He (Mr. Allen) had never been able to obtain the whole of the tin in solution, ignited stannic oxide being practically insoluble in hydrochloric acid.

Mr. HEHNER suggested that a still simpler solution might perhaps be arrived at by proceeding on the principle of a method for the estimation of mineral acids in vinegar, which was contained in a paper read before the Society some years previously by Messrs. Allen and Bodmer, viz., by distilling with sulphuric acid, carefully neutralizing the distillate (which would contain sulphate, chloride and acetate of sodium), igniting and titrating the alkalinity of the ash, which would give the acetic acid present in the distillate. He had never found any difficulty in qualitatively testing the ash of tinned meat. He simply boiled with hydrochloric acid, in which the stannic oxide was not soluble, and then with water, after which it dissolved. On the addition of sulphuretted hydrogen an abundant precipitate of sulphide of tin was often obtained.

Mr. A. C. CHAPMAN said that during the past two or three years he had had considerable experience in the separation of the sodium salts of certain organic acids from inorganic sodium salts by means of alcohol, and he had always found it a very difficult matter to effect a perfect separation. For technical purposes, however, the method would give quite satisfactory results, as was shown by the author of the paper.

The PRESIDENT suggested that the difficulty arising from the presence of nitric acid might possibly be overcome by using barium to catch the nitric acid; barium nitrate would be insoluble in alcohol. The subject of the separation of metals in small quantities under difficult circumstances was one that was constantly coming

forward in analytical laboratories, and it was evident that Mr. Allen had materials for a very interesting paper thereon.

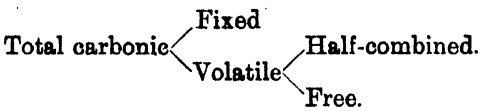
Mr. BLOUNT said that in the particular mixture under consideration the use of barium would lead to the formation of barium chloride, which would be too soluble in alcohol to render the modification practically applicable. He was afraid that in following Mr. Allen's proposition the process of distilling to a small bulk with sulphuric acid would involve the decomposition of the silver chloride.

THE ESTIMATION OF CARBONIC ACID IN NATURAL WATERS.

By CLARENCE A. SEYLER, B.Sc., F.I.C.

(Read at the Meeting, Nov. 3, 1897.)

THE carbonic acid of natural waters may occur in three forms: combined with alkaline earths or alkalies as mono-carbonates, half combined as bicarbonates, and fully-free carbonic acid. Of these the free and half-bound are approximately expelled on boiling, and hence I propose to call them the volatile carbonic acid. This is what is estimated by Pettenkofer's process:



The volatile carbonic acid used to be spoken of as free, but this name must be reserved for that which exists in excess of that necessary to form bicarbonates. The free acid is lost much more readily than the half-bound on exposure to the air.

Pettenkofer's method, though no doubt capable of accurate results with extreme care, is not fitted for the purposes of the busy analyst. It involves the use of many carefully calibrated vessels and frequent standardizing of variable solutions, and even at the best the results are subject to considerable uncertainty. On the assumption that the half-bound acid is equal to the combined, we may estimate the free carbonic acid by subtracting the combined from the volatile, as determined by Pettenkofer's process; but this is inaccurate with small quantities, as well as tedious. We require a rapid and accurate method of estimating the free carbonic acid independently. The proposals of Pettenkofer and Vignon to do so by a direct titration with lime or baryta water, using turmeric or phenolphthalein as an indicator, do not give satisfactory results. Trillich in 1890 appears to have been the first to give a satisfactory process by using sodium hydrate or carbonate, with phenolphthalein as indicator. I have used the process in regular practice since 1892; and in 1894, in the *Chemical News*, vol. lxx., p. 104 *et seq.*, I published the results of a careful examination of the accuracy of the process, together with the results of its application to different classes of water. The method does not seem to have attracted the attention in this country which I believe it deserves, though it is better known in Germany. My object is to direct the attention of analysts to the method, and to supplement my previous

papers with further experience of it. Referring to the journal already cited for details and explanation, I would say that the method depends on the exact neutrality of the bicarbonates, and the alkalinity of the carbonates, whether of the alkalies or alkaline earths, to phenolphthalein. Owing to the difficulty of keeping caustic soda free from carbonic acid, and also on account of its greater delicacy, I use a $\frac{N}{20}$ solution of sodium carbonate. To 100 c.c. of the water a few drops of a neutral alcoholic solution of phenolphthalein are added, and the liquid then titrated (best in a narrow vessel, with continual but not violent stirring) until the occurrence of a faint but permanent pink colour. This gives the free carbonic acid alone.* The combined acid is estimated on the same or a fresh portion of the water by means of methyl orange and $\frac{N}{20}$ acid. The volatile acid is the sum of the two titrations. If the water is neutral to phenolphthalein, it contains only bicarbonates, and no free carbonic acid. If a water is alkaline to the indicator, it can contain no free carbonic acid, and the volatile acid must be less than the fixed. This deficiency can be readily determined by careful titration with $\frac{N}{20}$ hydrochloric acid until the pink just vanishes.

Let m be the number of c.c. of $\frac{N}{20}$ hydrochloric acid required by 100 c.c. of the water, with methyl orange as indicator, p the number of c.c. of $\frac{N}{20}$ sodium carbonate required to produce the pink colour with 100 c.c. of the water, and p' the number of c.c. of acid required to discharge the pink if the water should be alkaline to that indicator. Then—

Free carbonic acid	...	...	1·1 p, or 1·1 $(p - 2m')$ parts per 100,000.
Fixed ,, ,,	...	...	1·1 m parts per 100,000.
Volatile ,, ,,	...	...	1·1 $(m+p)$, or 1·1 $(m-2p')$ parts per 100,000.
Total ,, ,,	...	...	1·1 $(2m+p)$, or 1·1 $(2m-2p')$ parts per 100,000.

Should the water contain free mineral acid, it will be acid to methyl orange, and can contain only free carbonic acid in addition. If m' be the number of c.c. of sodium carbonate required to destroy the pink with methyl orange, p being, as before, the amount necessary to produce the pink with phenolphthalein, then the free carbonic acid is given by the formula:

$$\text{Free } CO_2 = 1{\cdot}1\ (p - 2m').$$

Thus, in any case we can, by two simple titrations on 100 c.c. of the water, completely determine the amount and condition of the contained carbonic acid in a few minutes. The accuracy of the method has been confirmed in the best of possible ways both by myself and Kippenberger by comparison with Pettenkofer's method and gravimetrically.

The presence of ferrous salts in quantity requires a modification of the process, and in the case of highly-mineralized waters, such as the Hunyadi Janos water, some interfering factor remains to be explained; but such cases are rare. I give some examples of Kippenberger's careful comparisons of the methods, which show that the process may lay claim not only to practical, but to scientific, accuracy:

* If much free CO_2 is present, the experiment must be repeated, running in nearly the right amount at once.

Free CO_2.	Combined.	Sum.	Pettenkofer.	Gravimetric.
2·10	5·05	7·15	7·35	7·24
2·09	11·90	13·99	13·85	13·78
1·43	9·25	10·68	10·7	10·63
2·00	6·60	8·60	8·55	8·33
11·35	6·77	18·12	18·12	
5·67	3·38	9·05	9·06	

The formulæ in the case of waters alkaline to phenolphthalein have not been verified in this way, and must therefore be accepted with caution.

As the method depends for its accuracy on the exact neutrality of the bicarbonates to phenolphthalein, which has recently been challenged by Küster (*Zeit. f. Anorg. Chem.*, B. XIII., s. 127, October, 1896), I thought it necessary to repeat his experiments. Küster affirms that the neutrality of sodium bicarbonate is only approximate, this salt being slightly alkaline to the indicator. He states that on titrating a solution of sodium carbonate with an acid, the pink to phenolphthalein does not vanish exactly at half the quantity required with methyl orange, but much later, 2 per cent. more acid (reckoned on the methyl orange titration) being required. Although a loss of CO_2 during the titration of a $\frac{N}{2}$ solution in an Erlenmeyer flask would account for this phenomenon, he states that this cannot be the cause, since by "a somewhat careful working one can see no trace of carbonic acid being evolved from the solution," and he expresses his conviction that the explanation is a totally different one, lying in theoretical considerations which he elaborates at considerable length. This is certainly most unconvincing, and contrary to all my experience. In my experiments the carbonate was prepared in different ways: twice recrystallized and dissolved without ignition, ignited at a dull red heat, and also dried at 150° C. The carbonate was dissolved in well-boiled water, generally neutralized together with the solution of the indicator by means of a few drops of $\frac{N}{100}$ baryta, and made up to 100 c.c. The acid was added from a verified burette with a long capillary spit reaching nearly to the bottom of the tall narrow cylinder in which the liquid was titrated with continual stirring by a circular glass rod, which was never lifted above the surface of the liquid. In this way the formation of bubbles and loss of carbonic acid was as far as possible prevented.

The carbonate was also dissolved in enough water to make the solution $\frac{N}{2}$, and titrated by the same apparatus without further dilution, as also in an Erlenmeyer flask with the ordinary burette as carefully as possible. The following table gives the results:

Methyl Orange.	Methyl Orange × ½.	Phenol-phthalein.	Difference per Cent.	Remarks.
28·55	14·27	14·32	+·17	Soda twice recrystallized. Vol. 100 c.c.
28·57	14·28	14·27	−·03	
25·80	12·90	12·95	+·19	Ordinary soda ignited in muffle. Diluted.
25·85	12·92	13·00	+·27	
39·05	19·52	19·45	−·18	Twice recrystallized. Dried at 150° C.
22·85	11·42	11·60	+·70	,, ,, ,, ,, Not diluted.
28·55	14·27	15·20	+3·26	,, ,, Ordinary titration.

It is thus shown that, with proper precautions to avoid loss of carbonic acid, the pink of the phenolphthalein vanishes exactly at half the quantity required with

methyl orange within the limit of experimental error—that is, under 0·2 per cent. on the carbonate present. It is also shown that by working without dilution and other precautions, loss of carbonic acid of from 1 to 3 per cent. can easily occur, so that Küster's results are fully explained, and the neutrality of sodium bicarbonate vindicated.

As regards the utility of determining the carbonic acid in water, I would make the following observations: The interpretation of the results of water analysis, especially of the organic matter and nitrogen compounds, depends entirely on the origin of the supply—whether upland surface, mixed water from cultivated ground, deep or shallow well, the calcareous and pervious nature of the strata, etc. Now, the nature and amount of the carbonic acid is highly characteristic of waters from these different sources. In upland surface waters both the free and combined carbonic acid are characteristically low. In mixed water from cultivated land the free carbonic acid is low, and the combined generally moderate, while free carbonic acid over 0·5 per 100,000 is usually characteristic of water of underground origin. Its amount is, as a rule, greater in deep wells than in springs, and closely related to the porosity of the strata. Much fixed carbonic acid is also indicative of ground water, and related to the calcareous nature of the strata—a fact of importance in regard to the degree of nitrification. Many cases will occur to the experienced analyst in which these determinations will guide him in that most difficult task—the interpretation of the results of water analysis. Especially valuable in this connection is a determination of the free oxygen, which can now be rapidly and accurately made by the iodometric method of Ludwig Winkler. The amount of free oxygen is often distinctly correlated to the amount of free carbonic acid, varying in the opposite direction. Independently of these considerations, the carbonic acid is of importance in regard to the hardness of the water and the possibility of softening it, the free carbonic acid being as important in Clark's process as the combined and half-bound. Again, as regards the action on lead, the amounts of free and combined carbonic acid are, I believe, the chief factors.

Finally, I would draw attention to the not infrequent presence in natural waters of such as are alkaline to phenolphthalein, and therefore contain less carbonic acid than is required to form bicarbonates. The usual idea that a water which is strongly alkaline after boiling must contain alkaline carbonates is incorrect; the carbonates of lime and magnesia, especially the latter, are soluble as such in water, and exhibit an alkaline reaction with phenolphthalein. These alkaline waters are usually surface waters, or those containing magnesium carbonate, which have been exposed to the atmosphere. Sea-water is a typical example. I first met with this curious fact of the alkalinity of sea-water in the neighbourhood of Swansea, and my friend, Mr. J. E. Clennell, has had the kindness to test it for me at various points in the Atlantic, both north and south of the equator, with the result that the water was always alkaline to this indicator (strongly in the open sea, faintly near land), and therefore always devoid of free carbonic acid.

I had the opportunity of investigating a case of a fresh water of this kind in a reservoir supplied by a stream fed by springs, and considered this of sufficient interest to examine more closely. The streams entering and leaving, and the water of the

reservoir itself, were examined at intervals of a week, the temperature of the air and water and the degree of saturation with oxygen being also followed. This is of interest in connection with the fact previously observed by me, that water may be, and frequently is in Nature, supersaturated with oxygen to a very considerable extent; that is, may contain much more oxygen than is theoretically possible according to its coefficient of solubility at the temperature, even when exposing a large surface to the air. The temperature of the air was practically the same as that of the water at all depths. The stream entering contained a fair amount of free carbonic acid, and was generally nearly saturated with oxygen at the temperature of the air or water. In spite of this, on the first two occasions the water at the surface of the reservoir was alkaline to phenolphthalein and devoid of free carbonic acid. At the same time it was supersaturated with oxygen, the amount corresponding to a temperature some 5 degrees below that of the water. The water of the stream flowing out always contained a trace of free carbonic acid, and was practically saturated with oxygen at the temperature of the air and water.

On the following week the water of the surface had become acid to the indicator, containing about 0·4 parts per 100,000 of free carbonic acid, and was now deficient in oxygen, being saturated with the gas at about 5 degrees above the atmospheric temperature.

A week later the water had again become alkaline to phenolphthalein, and had reverted to a condition of supersaturation with oxygen to the extent of 7 degrees. A sample from a depth of 10 feet below the surface, however, contained at the same time as much as 0·7 parts of free carbonic acid, and was deficient in oxygen, being saturated at no less than 15 degrees above the temperature of the water, which was the same at that depth as at the surface. At the same time the distribution of chlorine and of combined carbonic acid was quite uniform, showing that the diffusion of gases must be much slower than that of salts in solution. The combined carbonic acid of the reservoir was considerably higher than that of the stream supplying it.

Discussion.

The President having invited discussion,

Dr. Rideal said he was sorry Mr. Seyler seemed to think that his paper in the *Chemical News* had not been read or appreciated. At the time of the publication of that paper he (Dr. Rideal) had taken note of it, and a student working in his laboratory at the time had repeated the work, and obtained satisfactory results. This mixed titration with methyl orange and phenolphthalein had proved useful to him in the examination of sewage-contaminated waters, in which the increased amount of carbonic acid was a measure of the amount of organic matter present in solution; it also threw a good deal of light on the variations in the chief constituents of sewage undergoing bacteriological treatment. Mr. Adeney, of Dublin, had some two years previously published a long monograph, in which the subject of the gases in water—viz., carbonic acid and oxygen—was dealt with at some length. Mr. Adeney had calculated the ratio of the oxygen to the carbonic acid much in the same way as Mr. Seyler had done in the case of the reservoir waters referred to in the

paper, and had drawn very important conclusions from his results. Mr. Adeney's task, however, had been a very laborious one, inasmuch as he had pumped out all the gases and analysed them by the ordinary methods of gas analysis, whereas the methods of estimating the dissolved oxygen and carbonic acid used by Mr. Seyler afforded a quick and ready means of arriving at the ratio between the two, and the conclusions were just as valuable as those obtained by the more laborious method. He might mention that only the previous day he had had to examine a mixture of caustic soda, sodium carbonate, and sodium silicate, and had got out his results by titrating with normal acid (using phenolphthalein as a first indicator, and finishing off the titration with methyl orange), calculating the total alkalinity in the two forms of sodium hydrate and sodium carbonate, and then determining the silica in another portion gravimetrically, calculating the percentage of sodium silicate, and deducting that from the alkalinity as sodium carbonate, thus getting the three determinations required. It had occurred to him, when Mr. Seyler was mentioning some of the methods he had been examining, that the discrepancy between the methyl orange titration and the amount of carbonic acid actually present might be due to the presence of sodium silicate in the mineral waters investigated.

Mr. BERTRAM BLOUNT inquired whether he was right in understanding that when the author said that sea-water was alkaline to phenolphthalein he meant that when neutral phenolphthalein was added a pink colour was obtained. If so, he thought that was a very interesting fact. It seemed to him that the practice of quoting analytical methods by the names of their authors rather than according to the principles upon which they were based was to be deprecated. It was almost impossible to carry in one's mind the multitude of names which it was thus rendered necessary to remember. The method, for instance, used in this investigation for the determination of oxygen was perfectly unfamiliar to him by the name of its author, though he gathered that it was of a similar nature to one (devised at an earlier period by Dr. Thresh) depending on the use of nitrous acid as a carrier for oxygen, which possessed the advantage that the danger of oxygen escaping during the process of titration into the neutral atmosphere above the water being titrated was removed. The time occupied was also much shortened. Unless some process of this kind were used, he did not think that oxygen determinations in water could be looked upon as possessing much real value.

Mr. ALLEN inquired whether the author had any actual evidence of the existence of bicarbonate of calcium. For his own part, he was very sceptical on this point. It was interesting theoretically, and no doubt convenient in practice, to make use of the expression "half-combined carbonic acid," but he had grave doubts as to the existence of such a body as bicarbonate of calcium. Some years ago a series of experiments had been made under his supervision, in which an attempt was made to demonstrate the existence of bicarbonate of calcium, but all that was arrived at was the fact that the proportion of carbonic acid requisite to keep the calcium carbonate in solution varied according to the extent to which the liquid was diluted. He raised this question from a theoretical point of view; it did not affect the practical accuracy of the results. He could fully endorse what Mr. Seyler had said as to the impracticability of precipitating the whole of the carbonate of calcium or carbonate of mag-

nesium in water by boiling. The amount which remained in solution was, however, very erratic.

Mr. HEHNER desired to reiterate Mr. Allen's remarks, which in fact comprised all that he himself wished to say. He also had made experiments with a view to ascertaining whether a neutral solution at ordinary temperatures could contain bicarbonate of calcium in the ordinary chemical sense, and had also failed to establish the existence of this body. He thought it might be probable, however, that at some temperature lower than the ordinary such a substance would exist. He did not himself quite see the object of going into the differentiation of the "half-combined" and fully-combined carbonic acid. With regard to the subject of dissolved oxygen, the diagram exhibited was exceedingly interesting. Might not the supersaturation with oxygen be due to algæ, which decomposed the carbonic acid and apparently liberated oxygen under the influence of light, since the phenomenon occurred near the surface.

Mr. A. C. CHAPMAN remarked that Mr. Allen and Mr. Hehner might equally extend their scepticism to the existence of calcium bisulphite, the evidence for the existence of both compounds being of an analogous character. In regard to the bicarbonate, the experimental results which had been obtained were not by any means conclusive as to its existence. A point incidentally referred to by the author, which was of some importance in connection with water analysis, and more especially so in the case of water containing a large proportion of alkaline chlorides, was the marked solubility of magnesium carbonate. In the analysis, for example, of London tertiary waters, which often contained a good deal of common salt, it was usual in the estimation of the alkaline carbonates to find considerable quantities of magnesium carbonate dissolved. The author's method would, of course, not apply to those waters (which, however, were not very numerous) in which organic acids of a peaty character were present.

Mr. RICHMOND said he remembered reading a paper in which it was shown that if a water saturated with oxygen at say 10° C. was cautiously raised to 15° C. without shaking or agitation, it would retain practically the whole of the oxygen, and be supersaturated. It had occurred to him on seeing Mr. Seyler's diagram that probably the water would be subjected to a good deal of agitation while running into the reservoir, and would absorb oxygen; this agitation would cease after the water had finished flowing into the reservoir, and a rise in temperature might conceivably take place unaccompanied by any diminution in the proportion of dissolved oxygen.

The PRESIDENT said that the interesting nature of the discussion in itself constituted a vote of thanks to Mr. Seyler, whom he would ask to kindly make such reply as was necessary.

Mr. SEYLER, referring to Dr. Rideal's remarks, said that he had read Mr. Adeney's paper with much interest, but the apparatus required was too expensive, and the operations too lengthy for ordinary use. He had given some examples of the application of the titrimetric method to sewage in his papers in the *Chemical News*, but confessed to some disappointment with the results in this case, as there were several interfering factors. Phenolphthalein was so much affected by ammonia that he was afraid the presence of this body in sewage might affect the results. This

difficulty might possibly be overcome by using tropæolin 000, which behaved exactly like phenolphthalein, but as far as he knew was not affected by ammonia. He had found large quantities of butyrates in sewage, and these were alkaline to methyl orange almost to the same extent as carbonates. It was quite true that the presence of silicates was a disturbing factor. In this connection it was of interest that Kippenberger had shown silicic acid to be alkaline to methyl orange—slightly, it was true, but distinctly. He (Mr. Seyler), however, thought that the difficulty was not of practical importance. With regard to the question raised by Mr. Blount as to Ludwig Winkler's method, he thought it was sometimes useful to refer to methods by their authors' names. In this method the source of inaccuracy mentioned by Mr. Blount was avoided, as in the process devised by Dr. Thresh, the oxygen carrier being, however, manganese hydrate instead of nitric oxide. The only objection to the method was the fact that sewage often absorbed iodine; but this could probably be avoided by titrating separately and deducting the iodine absorbed in this way. With regard to the scepticism which had been expressed as to the existence of calcium bicarbonate, he thought that the experiments on which such scepticism was based must have been very inconclusive. He thought that there could be no better proof of the existence of calcium bicarbonate than the figures which he had written down—the agreement would be impossible if calcium bicarbonate did not exist in the waters. Shortly before coming to town he had prepared a solution of calcium bicarbonate in the following way. He took a fairly strong solution of carbonic acid in water, and added lime-water until he got an indication of a precipitate of calcium carbonate, when he stopped the addition of lime-water, and filtered the solution (which was slightly alkaline to phenolphthalein owing to the solubility of calcium monocarbonate in water), and determined the volatile carbonic acid by Pettenkofer's method. Titration with methyl orange gave 20·7 milligrammes of carbonic acid per 100 c.c. Deducting the slight alkalinity (0·4 c.c.) for the monocarbonate present, there remained 20·3 as the amount of "half-combined" carbonic acid. Pettenkofer's method gave volatile carbonic acid 19·97. Thus, in a solution of calcium carbonate in carbonic acid neutral (like other bicarbonates) to phenolphthalein the volatile carbonic acid was shown to be approximately equal to the fixed, thus proving in his (the speaker's) opinion that the solution contained practically calcium bicarbonate. Very probably the solution on standing for some time would decompose, possibly into sesquicarbonate of calcium and free carbonic acid. There was no evidence of any abundance of algæ in the water supersaturated with oxygen. He thought that probably the explanation suggested by Mr. Richmond was the correct one. He would like to mention that the alkalinity of sea-water was enormously increased by the action of plant-life (marine algæ, etc.), which took up the "half-combined" carbonic acid, leaving magnesium monocarbonate in solution.

THE DETECTION OF GELATIN IN CREAM.

By A. W. Stokes.

(*Read at the Meeting, Nov.* 3, 1897.)

Very frequently, especially in summer, cream is thickened by the addition of gelatin to give it firmness and an appearance of richness. It is very difficult, if not impossible, to detect this by any method at present published. It is first necessary to precipitate the proteids entirely, for if any trace of these is left, this will come down afterwards and be quite undistinguishable from the gelatin. Unfortunately, most reagents which entirely precipitate proteids also at the same time bring down the gelatin.

The following method I have found to be perfectly successful, very minute quantities of gelatin being at once detected :

Dissolve some mercury in twice its weight of strong nitric acid (specific gravity 1·42); dilute this with water to twenty-five times its bulk. To about 10 c.c. of this solution add a like quantity of the cream and about 20 c.c. of cold water. Shake the mixture vigorously, leave it for five minutes, then filter. If much gelatin be present it will be impossible to get a clear filtrate. To the filtrate, or to a portion of it, add an equal bulk of a saturated aqueous solution of picric acid. If any gelatin be present, a yellow precipitate will be immediately produced. The whole operation is performed in the cold, and (if the mercury solution is ready) will not take more than ten minutes.

Picric acid will show the presence of one part of Nelson's gelatin (undried) in 10,000 parts of water. The same process would, of course, detect gelatin in milk, though I have never yet met with a sample adulterated in this manner.

A NEW "MILK-PRESERVATIVE."

By A. W. Stokes.

(*Read at the Meeting, Nov.* 3, 1897.)

Very lately a liquid called the "Rhodian Purifier" has been introduced for use with milk and other articles. It is claimed that a teaspoonful added to a pint of milk "will neutralize all disease germs—bacteria, microbes, bacilli, and other impurities—thus rendering it absolutely safe and pure, and keep milk sweet for several days in the warmest weather." This fluid was brought under the notice of several large milk-dealers, and from two of them was sent to me. I could find nothing in it but potassium nitrate and water, of the former of which there was a little over 19 per cent. Its preservative powers on milk were purely imaginary, since milk with and without this fluid (in the proportions mentioned) went sour equally soon in the month of August; while as for its "neutralizing germs," one sample was actually swarming with infusorial life, *Paramœcia* and other creatures equally large being fairly plentiful, as well as fungoid growths. After keeping the fluid for two months corked up in a

bottle, I found that the infusoria were all dead, but that living bacteria were very numerous, and also fungus spores.

The material is readily detected in the ordinary course of milk analysis. When the milk-solids are being incinerated, they deflagrate very prettily, and the ash, if taken up by a little dilute sulphuric acid, will give the nitrite reaction—*i.e.*, a blue colour with potassium iodide and starch; or in determining the fat by the Gerber, Leffmann-Beam, or Babcock process, a yellow colour is at once developed when the "preserved" milk mixes with the strong sulphuric acid. This colour, however, is not distinctive, but minute bubbles of nitrous gas are given off, which may readily be recognised by its odour. This evolution of gas makes the use of a closed tube, such as the Gerber tube, somewhat dangerous. An assistant of mine had three tubes, one after the other, burst in his hands in testing one of the samples of milk.

A NEW MILK-ADULTERANT.

By A. W. Stokes.

(*Read at the Meeting, Nov.* 3, 1897.)

Some few months ago an individual from Australia called upon a number of London milk-purveyors with a secret nostrum for concealing the addition of water to milk. He said that it had largely been used in Australia, and that 20 per cent. of water could by its use be added to milk without fear of detection. It proved on analysis to be water containing 1·96 per cent. of dextrin (British gum); it had a specific gravity of 1·0053,* and was to be added in the proportion of 4 ounces to the pint of milk. This certainly would not cover anything like 20 per cent. of added water.

As it contained a small amount of unaltered starch, it could at once be detected by the cautious addition of a solution of iodine. I have not heard of its actual use in England, but a case was quoted in a Sydney journal of a prosecution for the use of what was evidently this material, though from the evidence the analyst there was not quite clear as to the material used. I am aware that this use of British gum is not absolutely novel, but it certainly is unusual nowadays.

Discussion.

Mr. Richmond said that before making any other remark, he would like to express the pleasure he felt at seeing Mr. Stokes with the Society again. During the past two or three years Mr. Stokes had rarely been seen at the Society's meetings, and he (Mr. Richmond) felt sure that he was speaking the feeling of the meeting in expressing pleasure at seeing him. He also had met with the Rhodian preservative, and had made a full analysis of it, finding it to consist of a somewhat impure solution of potassium nitrate. He had also examined some other new preservatives, one of which contained potassium nitrate, salicylic acid, and sodium chloride, in about equal parts; another consisted mainly of a solution of salicylic acid in glycerin. With regard to this latter, it was stated that after a short

* The material having commenced to decompose, the actual specific gravity does not agree with the theoretical.

time it became impossible to detect its presence, although, as far as he knew, he had never failed to detect it in any sample containing it. He would like to ask Mr. Stokes whether he had attempted to apply his method to the estimation of the percentage of gelatin in cream. One of the first questions that a public analyst would be asked, if cross-examined in a case of this kind under the Sale of Food and Drugs Act, would be as to the proportion of gelatin present, and he (the speaker) thought there would be a considerable chance of a case falling through if the analyst was unable to say. There were, of course, a number of obvious methods by which the percentage of gelatin could be determined. It would probably be simplest to estimate the nitrogen in the filtrate from the mercuric nitrate solution, or it might possibly be done by polarization.

Mr. BODMER inquired whether, in testing for dextrin, it was necessary to coagulate the milk and test the serum, or whether operating on the original milk was sufficient to detect the quantity usually added.

The PRESIDENT said that Mr. Richmond had anticipated him in expressing pleasure at seeing Mr. Stokes with the Society again. It was a long time since Mr. Stokes had come to read a paper before them—almost as far back as the introduction of the Werner-Schmid process, which was very ancient history indeed.

Mr. STOKES said that he had not attempted to apply the method to quantitative estimations. Although the occurrence of gelatin in samples of cream was so frequent that the practice of adding it appeared to be systematic, its proportion was always very small. A mere fractional percentage was apparently sufficient to impart the apparent stiffness, which was the object of the admixture. In testing for dextrin, it was advisable to work upon the original milk, but care was necessary, as very little starch was left unchanged, and it was very easy to miss it if either too much or too little iodine were added.

ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

FOOD AND DRUGS ANALYSIS.

The Amount of Water in Raw Coffee. B. Niederstadt. (*Forsch. Ber.*, 1897, iv., 141.)—With the object of determining the average percentage of water in raw coffee as put upon the market, the author examined samples of several different kinds. These were dried to constant weight at 105° C., and gave the following results: Santos I., 13·33; Santos II., 12·20; Santos III., 11·23; Santos IV., 14·50; Bahia, 8·54; Menado, 10·72; Java, 10·75; Columbia, 10·00; Costa Rica, 10·62; Guatemala, 10·18; and Santos V., 9·45; the mean being about 11 per cent.

C. A. M.

The Composition of Potatoes. Balland. (*Jour. Pharm. Chim.*, 1897, vi., 298-300.)—The analyses of a large number of the principal varieties gave the following results expressed as percentages:

	Water.	Nitrogenous Substances.	Fat.	Sugars and Starches.	Cellulose.	Ash.
Normal state: Minimum ...	66·10	1·43	0·04	15·58	0·37	0·44
Maximum ...	80·60	2·81	0·14	29·85	0·68	1·18
Dried: Minimum	—	5·98	0·18	80·28	1·40	1·66
Maximum	—	13·24	0·56	89·78	3·06	4·38

The proportion of water is independent of the size of the potato, and of the variety, and appears to depend on the nature of the soil. The ash usually contains traces of manganese. The total acidity varies from 0·072 to 0·250 per cent.

C. A. M.

The Composition of Haricots, Lentils, and Peas. Balland. (*Jour. Pharm. Chim.*, 1897, vi., 196, 197.)—The following table gives the percentage results of the author's analyses:

	Haricots.		Lentils.		Peas.	
	Minimum.	Maximum.	Minimum.	Maximum.	Minimum.	Maximum.
Water	10·10	20·40	11·70	13·50	10·60	14·20
Nitrogenous substances	13·81	25·46	20·42	24·24	18·88	22·48
Fat	0·98	2·46	0·58	1·45	1·22	1·40
Sugars and starches	52·91	60·98	56·07	62·45	56·21	61·10
Cellulose	2·46	4·62	2·96	3·56	2·90	5·52
Ash	2·38	4·20	1·99	2·66	2·26	3·50

In the case of lentils, the Egyptian varieties are the richest in nitrogen, whilst the minimum of nitrogen and maximum of fat in the above table are found only in large Spanish lentils. Peas have the highest proportion of nitrogen before reaching maturity. Haricots, lentils, and peas all have about the same amount of acidity as beans, and they alter but little in their chemical composition even when kept for a long time. They absorb a considerable quantity of water—up to 100 per cent. of their weight in twenty-four hours, when first gathered, but this proportion gradually diminishes on keeping.

C. A. M.

On the Ash of Spices. A. Rau. (*Zeit. für öffent. Chemie*, iii., 439.)—A large number of samples of black and white pepper, cinnamon, pimento, and cloves was examined. The samples, first cleaned from earth and sand by sifting, furnished the following results:

Black Pepper.

212 samples were examined. The average ash amounted to 6·35 per cent., the maximum to 7·43 per cent.

43 samples contained between 5 and 6 per cent.
143 ,, ,, ,, 6 and 7 ,,
26 ,, ,, over 7 per cent.

Cinnamon.

142 samples were examined. The average ash amounted to 5·54 per cent., the maximum to 7·60 per cent.

30 samples contained between 4 and 5 per cent.
72 ,, ,, ,, 5 and 6 ,,
40 ,, ,, ,, 6 and 8 ,,

WHITE PEPPER.

155 samples were examined. The average ash amounted to 4·00 per cent., the maximum to 4·95 per cent.

60 samples contained between 3 and 4 per cent.
99 ,, ,, ,, 4 and 5 ,,

PIMENTO.

84 samples were examined. The average ash amounted to 6·38 per cent., the maximum to 8·35 per cent.

30 samples contained between 5 and 6 per cent.
53 ,, ,, over 6 per cent.

CLOVES.

78 samples were examined. The average ash amounted to 6·94 per cent., the maximum to 7·65 per cent.

45 samples contained between 6 and 7 per cent.
33 ,, ,, ,, 7 and 8 ,, H. H. B. S.

The Detection of Artificial Colour in Sausages. H. Weller and M. Riegel. (*Forsch. Ber*, 1897, iv., 204, 205.)—In the examination of a large number of different kinds of American sausages, the authors met with three samples which were of a suspicious colour, and from which the colouring matter could be extracted by digestion with glycerin and water, with alcohol, with amyl alcohol, and with ether, all the solvents being coloured from light red to deep red. On extracting the meat with acidified glycerin and water according to Bremer (ANALYST, this vol., 216), a bright red solution was obtained, but this, when evaporated on the water-bath after the addition of ammonia, remained unchanged. The coloring matter also dissolved readily in acidified amyl and ethyl alcohols, but on evaporating the solution in contact with wool fibre it was not possible to fix the colour in the wool, even in the presence of aluminium salts.

Since the sausages were strongly impregnated with salts, the authors considered it probable that the coloring matter was produced by the action of a salt on the blood-colouring matter, and their experiments confirmed this. It was found that flesh containing blood, when dried with sodium chloride, potassium chloride, sodium nitrate, sodium phosphate, or mixtures of these, yielded only minute traces of colour to the solvents. But, on the other hand, deep-red solutions were obtained from flesh containing blood which had been dried with potassium nitrate; and these behaved in the same way as the colored solutions obtained from the sausages.

From this it appeared that a chemical change of the oxyhæmoglobin had taken place during the drying with potassium nitrate, and this view was confirmed by the spectroscope, the acidified glycerin solution diluted with water giving a spectrum similar to that of methæmoglobin. After the addition of ammonium sulphide, a broad absorption band appeared at 52, and a narrower one at 64—evidently the spectrum of reduced methæmoglobin which has not hitherto been described. This

spectrum was identical with that of hæmoglobin, the formation of which was to be expected on treating methæmoglobin with ammonium sulphide.

It was further proved that the alteration of the oxyhæmoglobin was not caused by the action of the flesh fibrin brought into solution by the nitre, and it appeared to be a special characteristic of swine's blood hæmoglobin. It is well known that the oxyhæmoglobin of the blood of different animals differs considerably in composition, crystalline form, solubility, etc.; and this is in keeping with the fact that in one experiment in which calf's blood was dried with nitre, only very slight traces of colour could be extracted after two days' treatment with ether in a Soxhlet extractor. The fibrin from normal venous blood was readily soluble in a solution of nitre, whereas that from arterial or unhealthy blood, especially in the case of the ox, was frequently insoluble.

Hence it follows that the process recommended by Bremer (*loc. cit.*) can only be relied upon when it is possible to precipitate the colouring matter from its solution as a lake, and identify it chemically or spectroscopically. But since many vegetable colouring matters, which are soluble in water, cannot be precipitated in the form of lakes, whilst they are insoluble in amyl or ethyl alcohols, Bremer's method, like the others, may often fail. A microscopical examination, too, may be inconclusive when the sausage has been colored with an aqueous extract of a vegetable coloring matter thoroughly distributed, and in such cases the reactions given by the aqueous extract with iron chloride, lead acetate, calcined magnesia, manganese peroxide, sodium bicarbonate, etc., may give indications. The official method of the Berlin Police Council—viz., extraction with glycerin and water in the water-bath for fifteen minutes, or digestion in the cold with alcohol—is regarded by the authors as absolutely useless in the light of the experiments described above. C. A. M.

A Qualitative Test for Iodine in Cod-Liver-Oil and other Organic Preparations. A. Seyda. (*Zeit. für. öffent. Chemie*, iii., 359.)—Twenty grammes of cod-liver-oil are heated with 40 grammes of powdered sodium hydrate in a nickel basin for about eight hours, being meanwhile stirred from time to time with a spatula. The residue is dissolved in 400 c.c. of water in a beaker, and the particles of carbon filtered off by suction through a Watt's porcelain filter. The clear filtrate is then heated, and carbonic acid gas passed through it, until a trial portion, on treatment with excess of barium chloride, yields a neutral filtrate after boiling. The fluid is then cooled, and an equal volume of 96 per cent. alcohol added. The fine crystals of sodium carbonate which separate are removed by filtration through a Watt's filter, and washed three times with alcohol of 50 per cent. The alcoholic filtrate and washings are concentrated in a porcelain basin on the water-bath, the solution transferred to a platinum basin and cautiously evaporated, until a pure white residue is obtained. After cooling, this residue is dissolved in water, 5 c.c. of a very dilute solution (about 0·01 per cent.) of potassium nitrite and about 10 c.c. of chloroform are added, the mixture shaken, and finally treated with an excess of dilute sulphuric acid. The mixture is now again well shaken, and then allowed to stand for a few hours to settle clear. Finally, the chloroform is removed with a pipette and filtered

through a dry filter into a test cylinder for colour observation. If an equal quantity of pure chloroform be placed in another similar glass side by side with it, the violet colour (even if very faint) will be plainly discernible by contrast. H. H. B. S.

TOXICOLOGICAL ANALYSIS.

A New Method for the Determination of Chloroform in Post-mortem Examinations. A. Seyda. (*Zeit. für öffent. Chemie*, iii., 333.)—This is a colorimetric method based upon the well-known reaction of chloroform with soda and resorcin. The following solutions are prepared:

1. 1·4 grammes of pure chloral hydrate (corresponding to 1 gramme chloroform) is dissolved in 1 litre of water, and 100 c.c. of this solution are made up to 1 litre. This diluted solution will, therefore, correspond to 1 part of chloroform in 10,000, or 0·1 milligramme in 1 c.c.

2. A 10 per cent. aqueous solution of resorcin.

3. A 25 per cent. solution of sodium hydrate.

The determination is carried out as follows:

The chloroform having been extracted from the organs under examination by distillation in a current of steam, the distillate is made up to 50 c.c., and 10 c.c. transferred to a test cylinder, with 2 c.c. of resorcin solution and 1 c.c. of soda, and the whole heated to 80° C. by submersion in a water-bath.

Five comparative test solutions are now prepared in uniform glasses with 1, 2·5, 5, 7·5, and 10 c.c. of chloral hydrate solution respectively, whilst another is prepared with 2 c.c. of resorcin solution and 1 c.c. of soda. All these are made up to the same volume, and suspended in the water-bath. After heating for ten minutes, the glasses are removed from the bath, and the colours of the solutions compared.

H. H. B. S.

ORGANIC ANALYSIS.

A Characteristic Reaction of Cotton-Oil. G. Halphen. (*Jour. Pharm. Chim.*, 1897, vi., 390-392.)—In a former paper (ANALYST, xix., 283) the author stated that the zinc salts of the fatty acids of cotton-oil obtained from their solution in carbon bisulphide had a reddish-orange colour. He now finds that the same reaction is obtained by the action of carbon bisulphide on the oil itself, and that the production of the colour is accelerated by heat, by the presence of free sulphur in the sulphur chloride, and by the addition of a common solvent, such as amyl alcohol. The test is applied by mixing 1 to 3 c.c. of the oil under examination with equal volumes of amyl alcohol and sulphur chloride, containing about 1 per cent. of free sulphur, and immersing the test-tube in boiling brine for ten or fifteen minutes. If no red or orange colour is produced, another 1 c.c. of the carbon bisulphide is added; and if after five or ten minutes' heating the result is still negative, the addition of 1 c.c. is repeated.

The coloration is not of equal intensity with all cotton-oils, but varies according

to their previous treatment, though in all cases it is pronounced. By means of this reaction the author was able to detect 5 per cent. and less of cotton-oil in different mixtures. C. A. M.

On Illipè-Nut Fat. **H. Becker** (*Zeit. für öffent. Chemie*, iii., 545).—The fat expressed from the Illipè-nut has been imported into Europe from the Dutch East Indies for the past eighteen or twenty years in ever-increasing quantities. It is used principally in conjunction with palm-kernel oil in the manufacture of candles. A sample submitted to examination gave the following results:

Specific gravity at 100° C.	0·8854
Saponification number of the fat	194·04
Saponification number of the fatty acids	206·0
Iodine number of the fat	29·93
Iodine number of the fatty acids	31·64
Melting-point of the fat	35·5°–36·5° C.
Melting-point of the fatty acids	54·5°–55·5° C.
Solidification-point of the fat	24·6° C.
Solidification-point of the fatty acids	52·5° C.
Unsaponifiable	0·397 per cent.
Reichert-Meissl number	1·23
Free acid	17·245 per cent.
Mineral matter	trace

The fat was readily soluble in ether, partly soluble in cold alcohol, and readily soluble in hot alcohol, though separating out again on cooling. It was also readily soluble in chloroform, benzol, and petroleum spirit. H. H. B. S.

The Detection of Urobilin and Biliary Pigments in Urine. **E. Lepinois.** (*Jour. Pharm. Chim.*, 1897, vi., 389, 390.)—The detection of urobilin by itself is comparatively simple, but when the urine also contains biliary pigments, the results are much more uncertain. The usual method is to precipitate all the urinary pigments, with ammonium sulphate added just to the saturation-point of the liquid which has been slightly acidulated with sulphuric acid. The precipitate is collected and treated with absolute alcohol, and the solution thus obtained gives a spectrum characteristic of the pigment, and fluoresces on the addition of zinc chloride and ammonia in excess. Instead of this process, the author has for some time past used the following modification, which he recommends as more simple, delicate, and less tedious: 5 c.c. of a 10 per cent. solution of zinc chloride containing just sufficient hydrochloric acid to make the liquid clear are mixed with 20 c.c. of the urine, 3 to 4 c.c. of dilute ammonia (1 : 4) added, and the precipitate filtered off. The alkaline filtrate is fluorescent in the presence of urobilin, whilst the other urinary pigments remain on the filter more or less combined with zinc. In addition to the fluorescence, the solution will show the spectrum either of normal urobilin (urochrome) or of the abnormal urobilin of febrile urine.

Any biliary pigments present can be detected by mixing the precipitate with

several c.c. of water and dissolving it in acetic acid. This solution gives Gmelin's reaction with nitric acid containing nitrous acid as well as does the original urine.

The author objects to Deniges' recent proposal to use mercury salts as the means of separation, on the ground that it is less sensitive than the zinc chloride method, and that the solution of urobilin so obtained does not fluoresce. C. A. M.

Iodine Derivatives of Casein. **A. Siebrecht** (*Berichte*, 1897, p. 1824.)—Compounds of iodine with the numerous albuminoids and their immediate derivatives, the albumoses and peptones, can be readily formed by the following method.

Casein Periodide.—An intimate mixture of 80 grammes of casein and 20 grammes of iodine is gradually warmed on the water-bath under constant stirring. The resultant brown powder is then extracted with water-free ether in a Soxhlet apparatus for a few hours, when the iodine compound is left, which, after being air-dried, contains 17·8 per cent. of iodine. Its composition is, within very narrow limits, constant. Prepared in this way, casein periodide forms a yellow powder, little affected by cold water, but soluble in hot dilute alcohol, from which it is deposited in brown flakes as the solution cools. The compound can also be formed by boiling casein and iodine together in 70 per cent. alcohol; the casein dissolves, and on cooling the casein periodide is precipitated. A portion of the iodine appears to be in a loose state of combination; for, on treatment with an alkaline thiosulphate, the compound is decolorized, and after having been washed on the filter, dehydrated with alcohol and ether, casein iodide remains. This latter compound consists of a white powder, insoluble in the ordinary media. It has, like casein, the character of an acid, dissolving readily in an alkaline solution, from which, on acidulation, it is precipitated in an unaltered condition. Casein iodide contains phosphorus and sulphur, and can be distinguished from casein by its insolubility in sodium sulphite solution. It contains an average percentage of 5·7 of iodine.

Another compound, which the author terms "caseojodin," can be prepared by heating 100 grammes of casein periodide with 2 litres of 10 per cent. sulphuric acid for two hours on the water-bath. The reddish-brown powder formed is then filtered off, dissolved in a dilute alkaline solution, precipitated by the addition of acid, filtered off, and boiled with 70 per cent. alcohol, from which, on cooling, it is precipitated as a white, flocculent precipitate. After further purification it is obtained as a white powder, which contains, on the average, 8·7 per cent. of iodine. It is proposed to use this last preparation medicinally. W. J. S.

The Analysis of Gutta-percha. **J. A. Montpellier.** (*Rev. de Chim. Indust.*, 1897, viii., 300-302.)—Gutta-percha is obtained from the *Isonondras percha* in a similar manner to caoutchoue, and has more recently been prepared by extracting the leaves of the tree with a solvent such as benzene. It contains three proximate constituents, first isolated by Payen in 1851, to which have been given the names *gutta*, *fluavile*, and *albane*. The author gives the following scheme for its analysis:

Estimation of Gutta and Resins.—From 0·5 to 1 gramme of the finely-divided sample is placed in a weighed filter-paper, supported by a perforated platinum cone

which is suspended (its point in the liquid) in a flask connected with a Soxhlet apparatus. Here it is extracted with boiling alcohol for five or six hours, after which the cone is transferred to the Soxhlet extractor, and the extraction continued for the same length of time. The resins (fluavile and albane) dissolve in the boiling alcohol, whilst the gutta and impurities remain in the filter-paper, and are dried at 100° C. in a current of carbon dioxide and weighed. The impurities are determined separately and deducted.

Separation of Albane and Fluavile.—This can only be done incompletely, but an approximate determination of the amount of each may be made by extracting 5 grammes of the sample with alcohol as above, and evaporating the alcoholic extract until the albane begins to separate out. On cooling, the fluavile remains in solution, and the albane, which has crystallized out, is collected on a weighed filter, dried at a low temperature, and weighed.

Estimation of Water.—One gramme of the gutta-percha is dried at 100° to 110° C. in a current of carbon dioxide for six to seven hours.

Estimation of Impurities.—A weighed quantity (not exceeding 1 gramme) is extracted with boiling chloroform in the same way as in the determination of gutta with alcohol. This dissolves both gutta and resins, and the impurities left behind are dried in a current of carbon dioxide and weighed.

Ash.—This is determined in the usual manner. Its amount never exceeds 0·5 per cent. C. A. M.

The Volumetric Determination of the Nitro-Group in Organic Compounds. S. W. Young and R. E. Swain. (*Jour. Amer. Chem. Soc.*, 1897, xix., 812-814.)—The proposed method is based on the reduction of the nitro-group by means of stannous chloride, and titration of the excess of the latter with standard iodine, in accordance with the reactions

$$RNO_2 + 3SnCl_2 + 7HCl = RNH_2.HCl + 3SnCl_4 + 2H_2O,$$

and

$$SnCl_2 + 4I = SnCl_4 + SnI_4.$$

So far the author's experiments have been confined to dinitrobenzene, and further work is required to determine to what extent the method is generally applicable. The process was as follows:

Weighed quantities of pure dinitrobenzene were placed in Erlenmeyer flasks and dissolved in 10 c.c. of alcohol. These flasks were connected in series with a Kipp generator for carbon dioxide, and when filled with the gas the stoppers were removed, and sufficient stannous chloride solution introduced into each to reduce the dinitrobenzene and leave a slight excess. The current of gas was again passed through the flasks, which were heated on the water-bath until the reduction of the dinitrobenzene was complete. This was usually the case in one and a half to two hours. The addition of 20 to 25 c.c. of strong hydrochloric acid somewhat accelerated the reduction. The flasks were then disconnected, and the excess of stannous chloride in each titrated back with the standard iodine solution.

From the result the nitro-equivalent of the iodine solution was calculated, and

thence the percentage of nitro-group in the body. The following eight results were obtained in one series of determinations: 54·63, 54·48, 54·61, 54·40, 54·44, 54·49, 54·44, and 54·44 per cent., the theoretical percentage being 54·76.

C. A. M.

Direct Estimation of Chlorine, Bromine, and Iodine in Organic Substances. Separation of Bromine from Chlorine by Means of Permanganate. P. Jannasch and E. Kölitz. (*Zeit. anorg. Chem.*, 1897, xv., 68 and 66.)—The authors separate the halogens from the organic compound in the form of silver salts by either the Carius or lime method.

In whichever way the silver compounds are obtained, they are collected on a filter, washed, and, together with the paper, placed in a silver crucible, and fused with 5 or 6 grammes of pure caustic soda. The melt is extracted with warm water, the silver filtered off, the solution acidified with sulphuric or nitric acid, and the iodine, bromine, and chlorine finally separated from one another with thallium sulphate (I from Cl), or by the processes of distillation with acetic acid and sodium nitrite (I from Br and Cl), and with acetic acid and potassium permanganate (Br from Cl), which have been already described by Jannasch and Aschoff (*Zeit. anorg. Chem.*, 1892, i., 144, 245, and 248).

In another communication (*loc. cit.*, p. 66) the same authors call attention to the fact that this separation of Br from Cl by the action of acetic acid and permanganate cannot be effected in presence of much sodium acetate, as the latter body almost entirely prevents the evolution of free bromine. If, therefore, the original solution containing the halogens is strongly alkaline, it must first be neutralized with either nitric or sulphuric acid before the acetic acid required in the distillation is introduced.

F. H. L.

INORGANIC ANALYSIS.

Note on the Separation and Estimation of Lead, Copper and Arsenic. F. Jean. (*Ann. de Chim. Analyt.*, ii. [19], 361.)—The lead, copper and arsenic are separated from the metals of the second group thrown down in the usual manner by hydrogen sulphide. After washing the precipitate of the mixed sulphides by decantation, they are oxidized by digestion with warm sodium hypochlorite solution; the solution is then acidified with sulphuric acid, boiled to expel excess of chlorine, and the *lead* thrown down as sulphate by alcohol in the cold solution. The filtrate is boiled to drive off alcohol, sufficient ammonia added to redissolve the precipitate of copper arseniate first formed, and the *copper* titrated by sodium sulphide, the end point being indicated by the brown coloration produced by a drop of the liquid brought into contact with a drop of an alkaline solution of lead. The solution is then acidified with dilute hydrochloric acid and the copper sulphide removed by filtration; the *arsenic* is determined as arsenic acid by adding a few crystals of potassium chlorate, concentrating the solution to about 20 c.c., nearly neutralizing ammonia, adding 5 c.c. of sodium acetate, and titrating with uranium acetate

under the same conditions as are maintained in estimating phosphoric acid by the Joulie method.

By the above method 0·1212 gramme of lead was found in a mixture containing 0·1220 gramme, the whole of the copper (0·020 gramme), and 0·0370 gramme against 0·0375 gramme of arsenic was also detected. C. S.

Determination of Lead and Antimony in Tinfoil. A. Seyda. (*Zeit. für. öffent. Chemie*, iii., 364.)

I. *Lead Determination.*—Ten grammes of foil are dissolved in 25 per cent. hydrochloric acid with the aid of heat. If antimony is present to the extent of about 1 per cent., it will remain for the most part undissolved. The stannous chloride is then converted into stannic chloride by the addition to the hot solution of 25 per cent. nitric acid, which at the same time dissolves the remainder of the antimony. A slight excess of a 25 per cent. solution of soda is now added gradually to the clear fluid, which is meanwhile warmed and kept in motion until the precipitate at first formed redissolves, leaving only a slight opalescence. A small quantity of precipitated sulphur is then added to the solution, and hydrogen sulphide passed through it, until it acquires a distinctly yellow colour. After standing for twenty-four hours, the black precipitate is filtered off, and washed on the filter with solution of sodium sulphide, until a trial portion of the filtrate, after acidifying with hydrochloric acid, produces a pure white precipitate of sulphur. The washing takes a considerable time, but must be thoroughly carried out, or the tin will not be completely separated from the lead. The filter with the moist precipitate is now removed from the funnel, and carefully heated with nitric acid of 25 per cent., after which the filter is pressed together into a loose ball by the aid of a stirring rod and placed in the funnel, the nitric acid solution filtered several times through it until clear, and the ball of paper then washed, first with hot nitric acid, and afterwards with hot water, until a portion of the washings no longer gives a black precipitate with hydrogen sulphide. In this way, any lead sulphate that may be formed is completely removed from the filter. The filtrate is then transferred to a basin, about 20 c.c. of dilute sulphuric acid added, the liquid evaporated, and the lead sulphate filtered off through a porcelain Gooch crucible and weighed.

II. *Antimony Determination.*—The method proposed is an adaptation of Rose's: 5 grammes of the foil cut into strips are dissolved a little at a time in 100 c.c. strong nitric acid with the addition of 25 c.c. of water. The metastannic acid i washed out with nitric acid of 25 per cent. into a porcelain basin, the acid evaporat to dryness, and the residue dried in the water-bath. It is important that the perature at which it is dried does not exceed 100° C., as the anhydrides of tin and antimony do not appear to combine with soda. The dry powder while still warm is added a little at a time to 60 grammes of sodium hydrate heated to fusion, with which it is thoroughly mixed by means of an iron spatula. Combination takes place almost immediately. The fused mass is allowed to cool to about 100° C., and is then broken up and dissolved by the aid of heat in about 700 c.c. of water. The turbid fluid is cooled, transferred to a beaker, and mixed with one-third of its volume of alcohol of

96 per cent. After standing for twenty-four hours, the precipitate is filtered off by decantation, using a mixture of two volumes of alcohol and one volume of water for washing out the last portions from the beaker.

In place of washing the precipitate with dilute alcohol, which causes the loss of some of the antimony, a second fusion with sodium hydrate is resorted to. This is carried out as follows: The filter with the precipitate is dried at 100° C., the precipitate is detached and the paper cut up into strips, and added to 10 grammes of fused sodium hydrate, its destruction being facilitated by the addition of a little sodium nitrate. When the paper has disappeared, the precipitate is added to the fused mass, and the fusion, solution and filtration then proceeded with as before. In order to ensure the insolubility of the sodium antimoniate, the liquid should contain about 10 per cent. of sodium hydrate and 25 per cent. of alcohol. The antimony is finally weighed as Sb_2S_3. H. H. B. S.

The Analysis of Phosphor Bronze, Phosphor Copper, Phosphor Tin, etc. **M. Wickhorst.** (*Jour. Amer. Chem. Soc.*, 1897, xix., 396-398.)

Determination of Phosphorus alone.—One gramme of the sample is treated with 20 c.c. of aqua regia (HNO_3, 15 c.c.; HCl, 5 c.c.), and after the action has ceased water is added, then ammonia in excess, and the liquid is made up to 200 c.c. Copper, lead, etc., are precipitated with sulphuretted hydrogen, the liquid filtered, and the phosphorus determined in 100 c.c. of the filtrate by precipitating in the usual way with magnesia mixture and ammonia. In very accurate work the precipitate is dissolved in hydrochloric acid, and the magnesium ammonium phosphate reprecipitated, but for most purposes one precipitation is sufficient.

Complete Analysis of Phosphor Bronze.—Half a gramme of borings are heated with 5 c.c. of nitric acid, the residue rubbed well with a rod and filtered off after the addition of a little water, and washed with water containing a little nitric acid. The filter and its contents are ignited, and the weight of residue gives stannic oxide plus phosphorus pentoxide. The residue is then fused with 0·5 gramme of sodium carbonate and 1 gramme of sulphur in a covered crucible, and the excess of sulphur expelled over a Bunsen flame. The cooled mass is dissolved in hot water, excess of ammonia added, then 1 gramme of ammonium chloride, and when cold the phosphorus is precipitated with magnesia mixture. The precipitate is collected, washed with dilute ammonia solution containing a little ammonium sulphide, dissolved in diluted hydrochloric acid, reprecipitated, and the phosphorus determined in the usual manner. The difference between the phosphorus pentoxide calculated from the result and the residue of stannic oxide plus phosphorus pentoxide gives the amount of stannic oxide.

The treatment with nitric acid does not render the whole of the phosphorus insoluble with the tin. The small amount going into solution is determined by precipitating the filtrate of a duplicate sample with molybdate solution and estimating the phosphorus in the precipitate by one of the usual methods. This phosphorus is added to that obtained above. The filtrate from the stannic oxide plus phosphorus pentoxide is neutralized with ammonia, 5 c.c. of nitric acid added, then made up to 150 c.c. and transferred to a large platinum dish. In this a platinum foil about

2 inches square is suspended, and an electric current* passed through so as to precipitate the lead, as lead dioxide, on the dish, and the copper as metallic copper on the foil. The precipitates are washed well with water, dried, and weighed.

Iron and zinc may also be present. If so, the liquid is warmed, an excess of ammonium hydrate added, and then ammonium sulphide. The precipitate ignited gives iron oxide plus zinc oxide. When weighed it is dissolved in hydrochloric acid, the iron determined as hydroxide, and the amount deducted from the previous weight.

C. A. M.

A New Method for the Determination of Phosphoric Acid. Woy. (*Zeit. für öffent. Chemie*, iii. 321.)—In recurring to the subject of his recent papers on the determination of phosphoric acid as phospho-molybdic anhydride (*cf.* ANALYST, this vol., 250) the author remarks:

The methods for the precipitation of phosphoric acid by molybdenum, used up to the present, have been characterized, on the one hand, by the employment of a large excess of molybdic acid, and, on the other, by the abundant use of nitric acid. The reason for this is the peculiar behaviour of molybdic acid. It dissolves to a clear solution in water containing nitric acid, but separates out on the addition of ammonium nitrate, though the more nitric acid used, the less is the separation which takes place, in cold solutions. Consequently, strong nitric acid counterbalances the tendency of the ammonium nitrate to promote the separation of the molybdic acid. But if the precipitation is made in a strong nitric acid solution, the yellow precipitate becomes partly dissociated, the dissociation increasing or diminishing in proportion as more or less nitric acid is used. Hence, when much nitric acid is used, it is needful to employ a large excess of molybdenum to counteract the tendency to dissociation. Hundeshagen, however, has shown that, for the precipitation of phosphoric acid by ammonium molybdate, it is only needful for the molybdate to be present in the proportion corresponding to $1P_2O_5 : 24MoO_3$; and, further, that only a correspondingly small proportion of nitric acid and about 5 per cent. of ammonium nitrate are required.

Experiments based upon the foregoing considerations resulted in the adoption of a 3 per cent. aqueous solution of ammonium molybdate, which always remains clear, and may be relied upon at any time to contain the requisite proportion of molybdenum.

As working details of the method have already been published in this journal (*vide* abstract of author's former paper, above mentioned), it is only necessary to add that, as certain chlorides are found to be prejudicial to the precipitation, the author dissolves raw phosphates in sulphuric acid (taking care to prevent the gypsum formed from setting into a mass), afterwards neutralizing the portion of the solution taken with ammonia as far as is possible without causing a precipitate to fall.

H. H. B. S.

* Strength of current not given.—ABSTR.

Valuation of Phosphates according to their Citrate-Solubility. P. Wagner. (*Chem. Zeit.*, xxi., 905.)—In the official directions for working Wagner's method for the determination of the citrate-soluble phosphoric acid in Thomas phosphate, as accepted in Germany, it is stated as follows: "The phosphoric acid must be determined by the molybdate method; direct precipitation by magnesia gives high results when the percentage of silicic acid rises above a certain limit." It appears from experiments which have been recently carried out in the author's laboratory, that this obstacle to direct precipitation by magnesia has been overcome. The conclusions arrived at are as follows:

1. If the freshly-prepared citrate-extract of a phosphate be mixed with alkaline citrate solution and magnesia mixture, and stirred for the customary half-hour with the stirring machine, the precipitated ammonium magnesium phosphate will be found to be quite free from silica. It makes no difference whether the solution be filtered directly after the stirring is completed, or whether it be first allowed to stand for two hours.

2. Though the citrate extract of Thomas slag remains practically clear, however long it is allowed to stand, it nevertheless develops on standing a peculiar tendency to deposit silica on the addition of alkaline citrate, and this tendency increases from hour to hour, so that even with samples only moderately siliceous, a separation of silica takes place if the extract be allowed to stand for three hours before the addition of the alkaline citrate. When, however, alkaline citrate is added to freshly-prepared extract, no separation takes place, even if the mixture be allowed to stand for eight hours.

3. The tendency to the separation of silica is very considerably diminished by the precipitation of the phosphoric acid. In a citrate-extract three hours old, no silica was separated when the alkaline citrate and magnesia were added together and the mixture allowed to stand for two hours.

4. It follows: (*a*) that the precipitation by means of magnesia mixture should be made, if possible, in freshly-prepared extract, and (*b*) that to ensure the non-separation of silica, especially when the extract is not quite fresh, a mixture of alkaline citrate and magnesia mixture should be used to precipitate the phosphoric acid in the extract.

The mixture of alkaline citrate and magnesia mixture is prepared as follows: 200 grammes of citric acid are dissolved in ammonia solution of 20 per cent., and the solution diluted to 1 litre with ammonia of the same strength. The litre of alkaline citrate so prepared is mixed with 1 litre of the official magnesia mixture.

For the determination of the citrate-soluble phosphoric acid, 50 c.c. of extract, either freshly prepared or not more than one hour old, are mixed with 50 c.c. of the above precipitating-fluid, and the mixture stirred for half an hour with the stirring machine. If the precipitate is not filtered off immediately, it should, at any rate, not be left standing longer than one hour.

The author has submitted the above modification of his process to the consideration of the chemists of the German experimental stations (*cf.* ANALYST, April to July, 1896). H. H. B. S.

REVIEW.

THE PRINCIPLES AND PRACTICE OF BREWING. BY WALTER J. SYKES, M.D., D.P.H., F.I.C. (London: Charles Griffin and Co., Limited, 1897.) Price £1 1s.

The appearance of a work such as this serves to remind one of the enormously rapid advances made during recent years in our knowledge of the scientific principles underlying the brewing processes. On glancing through Dr. Sykes's new book, the first feeling, perhaps, is one of astonishment at the vast number of investigations which have been undertaken by men of science with the object of throwing light upon the complex changes involved in the manufacture of beer, investigations which have in some cases earned for their authors world-wide renown in scientific circles. In no other ancient industry, probably, has the transition from the darkness of empiricism to the light of science been so rapidly effected. The author rightly commences his work with a discussion of the physical principles involved in brewing operations, a matter which is frequently ignored in treatises dealing with the science of brewing. To a considerable number of brewers the expressions *density*, *specific heat*, and *latent heat*, convey no definite and clear meaning, whilst the real nature of the thermometer, and the relation between the two chief thermometric scales, are far from being clearly understood. To such Dr. Sykes's clear exposition will prove interesting and useful. The second chapter deals with the chemistry of those substances with which the brewer is chiefly concerned, and includes a short account of the construction and use of two forms of polarimeter which are best adapted for the examination of brewing materials—namely, the Laurent instrument and the half-shadow polarimeter made by Schmidt and Haensch. In this section an account is given of certain important groups of organic compounds, such as the alcohols, aldehydes, ketones, hydrazones, etc., a knowledge of which is necessary to the understanding of the constitution of the sugars. It is matter for regret that the scientific training with which the average brewer commences his brewing career is such as to render this account, clear as it is, somewhat beyond his grasp. To many, however, the addition will be very welcome. A full description of the more important carbohydrates is given, and includes an accurate account of all the most recent investigations, whilst the better known proteids and enzymes receive an amount of attention which is justified by the all-important parts they play in practice. As a minor point in nomenclature, it would appear better to employ the word "glucose" only in speaking of the commercial product, whilst referring to the carbohydrate itself as "dextrose," the former word being at the present time somewhat indefinite. It may be remarked, in passing, that the analysis of commercial glucose by Birneisel quoted on page 80 can scarcely be taken as representing the average composition of the sugar manufactured in this country. It appears to be what is often euphemistically described as a "commercial analysis" in which the presence of maltose is ignored and the increased cupric-reducing power on hydrolysis attributed solely to dextrin, which is, as a rule, present only in traces. An admirably clear account of the numerous researches which have been undertaken with the object of elucidating the changes involved in the hydrolysis of starch at the instance of diastase is given, for which alone Dr. Sykes would well deserve the thanks

of all chemists desirous of knowing to what extent this most difficult problem has been solved. A description of the hop-plant and of the various constituents of the strobiles follows. The first chapter of the second part deals with that instrument which may be described as the sheet-anchor of the modern brewer—the microscope. A short account of its various parts is given, together with some useful hints to intending purchasers, instructions for its use, and a lucid description of the simpler bacteriological methods. Perhaps no section of this work is deserving of greater praise than that treating of vegetable biology and fermentation. Starting with a description of the *Protococcus pluvialis*, as a unicellular organism which may conveniently be used for the purpose of illustrating certain important biological functions, such as *assimilation, respiration*, and *reproduction*, the author passes on to a consideration of the life history of the yeast organism, the torulæ, certain bacteria, and some common mould fungi. To describe clearly and concisely everything of importance that has been thought and done in connection with the decomposition of sugar by the yeast organism is no easy matter, and Dr. Sykes is to be congratulated on the success with which he has accomplished the task. As far as possible in chronological order, and with a due appreciation of the importance of each, one finds all the chief theories of fermentation discussed, together with an account of the many important chemical and biological investigations which led up to the brilliant work of Hansen on the production of single-species yeast and its use on a commercial scale. A short account of the structure of the barley-grain, and of the nature of the interesting changes occurring during the process of germination, is also given. The first chapter of the third part deals with the composition of natural waters and their suitability for the production of different classes of beers, the remainder of the book being devoted to the subjects of malting and brewing, considered in their more technical aspects. The chapters dealing with the construction and arrangement of brewery-plant and with the practice of brewing are such as to add greatly to the value of the book.

A chapter on the ills to which beer is heir, and five short appendices (one of which deals with Buchner's recent work on alcoholic fermentation without yeast-cells), bring this work to a conclusion. A description of the methods adopted for the analysis of brewing materials and products has been omitted, as it would, to use the author's own words, "have made the book too voluminous," and constitutes in itself "a subject sufficiently large to form a separate treatise." A careful perusal of the work under review leads one to hope that Dr. Sykes will one day give us a companion volume dealing with this branch of the subject. Every page bears testimony to the fact that no pains have been spared to render this work as complete as the present state of our knowledge permits. It will undoubtedly be of the greatest assistance, not merely to those for whom it is professedly written, but to all chemists and biologists who are interested in the numerous scientific problems which the fermentation industries present. To such it will prove not only a valuable work of reference, but will serve the still more useful purpose of directing attention to many obscure points which yet call for experimental study. The illustrations are well executed, and a complete table of contents and a good index add to the usefulness of the book.

A. C. C.

ERRATA.—Page 282, lines 17, 19, and 20, *for* "per cents." *read* "parts per thousand."

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THE ANALYST.

JANUARY, 1898.

PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS.

THE monthly meeting of the Society was held on Wednesday evening, December 1, in the Chemical Society's Rooms, Burlington House, the President (Dr. Bernard Dyer) occupying the chair.

The minutes of the previous meeting were read and confirmed.

The President read the following list of officers for the ensuing year, as proposed by the existing Council for election at the annual meeting:

President.—Bernard Dyer, D.Sc.

Vice-Presidents (who have filled the office of President).—M. A. Adams, F.R.C.S.; A. H. Allen; Sir Chas. A. Cameron, M.D., F.R.C.S.; A. Dupré, Ph.D., F.R.S.; Otto Hehner; Alfred Hill, M.D., F.R.S.E.; J. Muter, M.A., Ph.D., F.R.S.E.; Thos. Stevenson, M.D., F.R.C.P.

Vice-Presidents (who have not filled the office of President).—A. P. Aitken, D.Sc., F.R.S.E.; W. W. Fisher, M.A.; J. A. R. Newlands.

Hon. Treasurer.—E. W. Voelcker, A.R.S.M.

Hon. Secretaries.—E. J. Bevan, Charles E. Cassal.

Other Members of Council.—Bertram Blount, Sidney Harvey, C. G. Moor, M.A., F. Wallis Stoddart, Walter J. Sykes, M.D., John White, W. C. Young.

The names of those members of the Council who do not retire this year are R. Bodmer, A. Wynter Blyth, M.R.C.S., A. C. Chapman, S. Rideal, D.Sc., J. E. Stead, J. A. Voelcker, M.A., B.Sc., Ph.D.

Mr. Leonard K. Boseley, analyst to Messrs. James Keiller and Sons, of Silvertown, was proposed for election as a Member of the Society, and Mr. — O'Shaughnessy, assistant to Mr. H. Droop Richmond, as an Associate.

The following papers were read:

"Milk Analysis and Certification." By James Edmunds, M.D.

"A Method of Estimating Tannic Acid by Means of the Polarimeter." By R. F. Wood-Smith.

"A Butter Eighteen Years Old." By E. G. Clayton.

"Note on Mineral Matter in Commercial Bleached Ginger." By E. J. Bevan.

The following gentlemen were elected members of the Society: Mr. S. H. Collins, F.I.C., Assistant Agricultural Chemist to the Indian Government; Professor James Hendrick, B.Sc., F.I.C., the University, Aberdeen; Mr. W. H. Jackson, Analyst to the West Yorkshire Coal and Iron Company; and Mr. E. H. Roberts, Analyst to the Dairy Supply Company, Ltd.

On the proposal of Mr. HEHNER, seconded by Mr. CHAPMAN, Mr. John Hughes and Mr. Bertram Blount were appointed auditors of the Society's accounts for the year.

The PRESIDENT announced that, although the Annual Dinner would take place on Tuesday, January 18, the Annual Meeting, for the election of officers, etc., would not take place until February, when it would be held in conjunction with the ordinary meeting.

AN IMPROVED MILK-SCALE.

BY H. DROOP RICHMOND.

(*Read at the Meeting, Nov.* 3, 1897.)

WHEN determining the density of milk, it is frequently a matter of convenience to make the estimation at the temperature at which the milk happens to be, instead of adjusting it to the normal temperature. When this is done, it is necessary to make a correction to ascertain the value at 60° F.

Vieth has published a set of tables (ANALYST, x. 70) founded on those of Fleischmann, and these I have found correct; numerous experiments have shown that the difference between the density at 60° F. shown by Vieth's tables, and that actually determined, very rarely exceeds 0·0002.

Seeing the favour with which slide rules are regarded at the present time, it has long been my desire to incorporate a corrector of specific gravity with the "milk-scale." I have now succeeded in doing this. On the body of the instrument the temperature is engraved, each degree of temperature being of dimensions corresponding to the increase of specific volume (specific volume of milk at 32° F. = 1); on the slide the scale of specific gravity is marked, each degree having a value inversely proportional to the increase of expansion with the specific gravity. To use the instrument, the specific gravity found (*i.e.*, the apparent specific gravity determined in glass vessels) is placed against the line 60° F. (marked with an arrow); the true specific gravity is read off against the temperature at which the determination was made.

The "milk-scale" is now made by Messrs. Baird and Tatlock, London, with the addition of the specific gravity corrector.

DISCUSSION.

The PRESIDENT said that very many analysts were already much indebted to Mr. Richmond for the time saved in calculation by the use of his milk-scale, and the introduction of the modification now described would add to their obligation.

Mr. A. W. STOKES said that the working of the scale was greatly facilitated by the use of a simple index, made by bending a flat piece of metal, a plan which he had found to answer better than any of the other devices made, which were more or less complicated.

The PRESIDENT said that an ingenious little apparatus of a nature somewhat similar to that mentioned by Mr. Stokes had been introduced some time previously

by Mr. Cassal. Among the advantages of such a device was that it prevented the temptation to make ink-marks on the scale.

[NOTE.—As the question of pointers was raised in the discussion, I may point out that the first was used by Mannheim nearly fifty years ago on the slide-rule. It was first applied to the milk-scale by A. E. Johnson, the most recent improvement being the use of a vernier instead of a pointer, as proposed by Sykes.] H. D. R.

NOTE ON THE GRADUATION OF LEFFMANN-BEAM BOTTLES.

BY G. E. SCOTT-SMITH AND A. B. SEARLE.

(Read at the Meeting, Nov. 3, 1897).

IN estimating the amount of fat in milk, we have recently been much perplexed by the occurrence of a great discrepancy between the results of the Leffmann-Beam and Werner-Schmid processes. This was the more surprising as the two methods had given us very concordant results in the past.

We first directed our attention to the reagents used. The amylic alcohol was purified by shaking with brine to remove any ethylic alcohol which might be present. It was then allowed to stand for twenty-four hours over potassium carbonate, and was then distilled. That portion distilling between 128° C. and 131° C. was used for the experiments. The sulphuric acid was found to be of the specified strength as calculated from the specific gravity (1·843). Using these purified reagents no better results were obtained.

A mixture of amyl alcohol and hydrochloric acid some weeks old was compared with a mixture prepared from the same reagents a few minutes before use. Experiments were made at the same time in which the quantity of sulphuric acid used was slightly varied, a constant volume of 15 c.c. of milk being used in each case. The volume of fat thrown up was noted after whirling one, two, three, four, and five minutes respectively, with the following results:

	Fat ; per Cent.				
	In one minute.	In two minutes.	In three minutes.	In four minutes.	In five minutes.
Using 9 c.c. H_2SO_4 and "old" amyl alcohol mixture	3·7	3·3	3·3	3·3	3·3
Using 9 c.c. H_2SO_4 and "new" amyl alcohol mixture	3·7	3·3	3·3	3·3	3·3
Using 10 c.c. H_2SO_4 and "old" amyl alcohol mixture	3·7	3·3	3·3	3·3	3·3
Using 10 c.c. H_2SO_4 and "new" amyl alcohol mixture	3·7	3·3	3·3	3·3	3·2

After whirling for one minute the separation was not sharp, as the layer of fat contained a large quantity of flocculent matter. After two minutes' whirling the separation of the fat was almost complete, and after three minutes the separations were always sharply defined. These results are satisfactory, in so far as they show

that the amount of sulphuric acid can be varied to a moderate extent without affecting the accuracy of the results.

Being convinced that our reagents were pure and that our method of procedure was correct, we attempted to find whether the source of error lay in the graduations of the bottles. We were aware that attention had been directed to the fact that it was necessary to calibrate the bottles before use, but we could not find a ready method for effecting this. As the 80 divisions on the neck of the bottle represent collectively a volume of only 1·2 c.c., the calibration is attended with difficulties which are not met with in many other cases.

We first attempted to calibrate the bottles by running in water from a fine burette, but this method was found to be useless, as an error of ± 0·05 c.c. per 10 divisions corresponded to 0·3 per cent. of fat, and in practice an error of this magnitude was unavoidable, chiefly owing to the tendency of the water to adhere to the sides of the neck of the bottle.

In later experiments proof-spirit was used, and the liquid weighed instead of being measured. In each case the bottle was filled up to the lowest division (number 80) with the spirit, and weighed carefully. More spirit was then run in from a small pipette until the liquid stood at the 70th division, when the bottle and its contents were again weighed, and this procedure was repeated at each 10 divisions until the liquid had reached the zero-mark of the bottle. This method appears to provide a ready and fairly rapid method of checking the accuracy of the graduations. It overcomes completely the difficulty due to adhesion of the liquid to the neck, without the inconvenience and necessity of whirling, with consequent liability to change of temperature and correction for expansion. If a balance turning to half a milligramme be used and the calibration conducted expeditiously, the loss of alcohol by evaporation is insignificant, and the use of mercury, with its objections, is avoided, the slightly greater accuracy obtained by the use of the latter being little or no advantage compared with the greater tediousness of the method. If the temperature of the balance-room is constant, no correction need be applied, as only the relative volumes of the graduations are required. The following are some of the figures obtained:

Weight of spirit in grammes:	1.	2.	3.	4.	5.	6.	7.	8.
80 to 70 divisions	·114	·117	·144	·141	·156	·155	·134	·150
70 to 60 ,, ...	·095	·150	·145	·149	·145	·150	·145	·129
60 to 50	·107	·102	·158	·142	·142	·150	·151	·161
50 to 40	·141	·119	·143	·131	·150	·151	·145	·145
40 to 30	·145	·149	·150	·134	·150	·134	·144	·157
30 to 20	·148	·153	·143	·150	·150	·166	·160	·157
20 to 10	·151	·147	·153	·142	·150	·151	·150	·148
10 to 0	·142	·157	·150	·158	·150	·146	·156	·150
Total weight of alcohol (in grammes)	1·042	1·099	1·186	1·147	1·193	1·203	1·185	1·197

These figures show how very imperfectly the bottles are graduated by the manufacturers, and how necessary it is to check each bottle before use if the Leffmann-Beam process is to be relied on. Thus, if No. 6 bottle were used, and the reading taken between divisions 80 and 50, the amount of fat shown would be 2·7 per cent.,

instead of the 3 per cent. actually present in the milk. In the case of bottle No. 1, if the reading were taken between the 50th and 80th divisions, a milk containing 2·3 per cent. of fat would show 3 per cent.

Bottles Nos. 3 and 5, on the other hand, are much more evenly graduated than any of the others in this series, and these two bottles have been found in practice to give fairly good results. Another set of bottles, calibrated in the same manner as the first set, showed that the scale in each was very uniformly graduated, but all the divisions were slightly too large. Hence the percentage of fat indicated was rather less than the true amount present, but the error was not sufficiently great to prevent the bottles being used.

Discussion.

Mr. A. W. Stokes agreed with the authors that the best liquid to use was alcohol, which possessed, among other advantages, that of being able to pass easily down a very narrow tube. He thought, however, that in filling the bottles with a pipette an error might arise through drops of liquid clinging to the sides of the tube, unless care was taken to get rid of such drops by rotating the bottle after the liquid had been added.

Mr. Richmond thought that alcohol was not a desirable fluid to use on account of its sensibility to changes of temperature. No provision seemed to have been made by the authors for obtaining a constant temperature. He thought it ought to be constant to a tenth of a degree, as otherwise the expansion or contraction of the alcohol would give rise to a distinct error. He had himself obtained excellent results with water. As far as he could remember, the bottle No. 3 was exactly correct, according to a specification which he had drawn up for Messrs. Baird and Tatlock, and to which all bottles supplied by that firm were made. He had found that a satisfactory method of calibrating these bottles was by means of a finely divided rule. He had not himself used the Leffmann-Beam process at all for about two years, but had previously used about two or three hundred of the bottles, and had always found them to be excellently graduated. He could not say whether they had fallen off in character; but from the results shown in the paper this would seem to be the case.

Mr. Hehner said that faulty graduation was not found merely in Leffmann-Beam bottles, but in almost every other kind of measuring instrument. He had lately checked the graduation of a number of burettes, and had found that out of a dozen, only three came up to the requirements of an ordinarily careful analyst, the other nine being from 1 to 1·7 per cent. out. Too much trust had been put in the makers of scientific instruments in this matter, and he certainly thought that every burette and pipette should be checked before being used, as had been suggested by Mr. Richmond years before.

The President said that he had no experience of Leffmann-Beam bottles, but he had used a set of Gerber bottles for a long time, and had never met with any discrepancies such as would indicate inaccurate graduation. Occasionally the results did not agree very closely, but this had generally been due to imperfect whirling before the use of the apparatus had been thoroughly mastered. It was very whole-

some indeed for papers of this kind to be brought before the Society. Most analysts had had experiences similar to those which Mr. Hehner had mentioned, and had grown cautious in accepting the graduation of the instrument makers. It was not merely in instruments for the measurement of capacity that errors were found. They were very common indeed in hydrometers, even of a fairly expensive kind, and even in thermometers, which were so very easily tested.

ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

FOODS AND DRUGS ANALYSIS.

The Determination of Fat in Watered Milk, Human Milk, Humanized Milk, and Condensed Milk. J. Froidevaux. (*Jour. Pharm. Chim.*, 1897, vi., 485-489.)—In the author's opinion, the most exact method of determining fat in milk is to precipitate the fat, together with the casein, by means of a dilute acid, and to extract the precipitate with ether But in the case of the milks cited above, coagulation is incomplete and clear filtration not possible. The author accounts for this by the fact that normal cow's milk contains a large amount of soluble calcium salts (from 6 to 7·5 grammes per litre), chiefly phosphates, which largely assist the coagulation; whereas human milk only contains from 1·5 to 2·8 grammes per litre. In the case of humanized milk the cause is the same, as the phosphates are precipitated by the sterilization, and only partially redissolve on cooling. Similarly, condensed milk, when diluted, according to the directions, with four or five parts of water, contains only 3·5 to 4 grammes of mineral matter per litre. Experiments showed that the added sugar played no part in the imperfect coagulation, which was entirely due to the small proportion of salts.

On these facts the author has based the following process, which he has found rapid and exact, and applicable in every case. It consists in precipitating the casein and fat with a solution prepared by mixing about 70 grammes of freshly precipitated calcium phosphate with 12 c.c. of glacial acetic acid and about 250 c.c. of water, and making the solution up to 2 litres. In the determination 90 c.c. of this solution are mixed with 10 c.c. of the milk, the precipitate filtered off, dried, and extracted with ether in a Soxhlet apparatus. If required, the lactose may be determined in the filtrate in the usual way, after adding a few drops of potash to facilitate the precipitation of the cuprous oxide.

In the case of condensed milk, 10 grammes are diluted with warm water, the mixture cooled, made up to 100 c.c., and 10 c.c. taken for the estimation. C. A. M.

Action of Micro-organisms of Milk on Indigo-Carmine. L. Vaudin. (*Ann. de Chim. Analyt.*, vol. ii., pp. 441-445.)—From a series of experiments made on the decolorization of indigo-carmine by milk, the author agrees with Duclaux in

attributing this reaction to the influence of microbes, and not to the action of albuminoids. He suggests that the addition of alkalies and antiseptics to milk should be prohibited, and that the decolorization test should be applied to all milk offered for sale, the minimum permissible standard of time ensuing before decolorization occurs being fixed at twelve hours at temperatures below 15° C., eight hours at 15° to 20° C., or four hours above 20° C. C. S.

Estimation of Volatile Fatty Acids in Butter. E. Wrampelmeyer. (*Landw. Versuchsst.*, 1897, xlix., 215; through *Chem. Zeit. Rep.*, 1897, 269.)—In carrying out the Leffmann-Beam process (*cf.* ANALYST, xxi., 259), the author dissolves the butter soaps in 250 c.c. of well-boiled hot distilled water, adds one drop of indicator and 50 c.c. of sulphuric acid (strength not stated). The whole is then distilled in a current of superheated steam, which is introduced into the flask through a tube reaching below the level of the liquid; and the vapours are made to pass through a condenser at least 0·5 metre long. The steam is obtained from distilled water free from CO_2, and is superheated by means of a flat flame burner during its passage along a copper tube 30 cm. long and 1·4 cm. in diameter. By working in this manner, the amount of decinormal alkali required by 5 grammes of the butter-fat is increased some 5 or 6 c.c., and as the figures yielded by pure margarine remain the same as in Wollny's process, the delicacy of the test is thus raised 15 or 20 per cent. F. H. L.

On the Compulsory Addition of Sesame-Oil to Margarine. von Raumer. (*Zeit. angew. Chem.*, 1897, 749-751.)—By an imperial statute of July 4, 1897, it was made obligatory on all margarine manufacturers in Germany to add at least 10 per cent. of sesame-oil to their products, so that any subsequent admixture of the margarine with butter might be readily detected by Baudouin's reaction.

In the author's opinion, such an addition is absolutely useless, for, apart from the fact that the margarine and butter industries are frequently carried on by the same individuals, and that the greater part of the admixture of margarine with butter takes place in large factories, where obviously margarine containing sesame-oil would not be used for the purpose, it is pointed out that Baudouin's test cannot always be regarded as conclusive. Thus it was shown by Spampani and Daddi (*Staz. Sper. Agric. Ital.*, 1896, xxix.) that the fat from the milk of goats whose fodder had contained sesame-oil gave Baudouin's reaction.

Of various vegetable coloring matters examined by the author, only one—turmeric—gave the same colour as sesame-oil with hydrochloric acid and furfural. But although turmeric gave the pink colour with hydrochloric acid alone, it was not possible to subsequently test for sesame-oil by adding furfural. (*Cf.* ANALYST, this vol., p. 235.)

In the case of yellow and orange coal-tar colours, the following gave a pink coloration with hydrochloric acid alone: (1) Ponceau. (2) Orange II., β-naphthol orange, tropæolin 000 No. 2. (3) Acid yellow G, the sodium salt of amido-azo-benzene sulphonic acid. (4) Dimethyl aniline orange, the sodium salt of dimethyl-

amido-azo-benzene-mono-sulphonic acid. (5) Methanil yellow S, the sodium salt of a disulphonic acid of phenyl-amido-azobenzene.

In the case of the two first, it is suggested that the colour might have been due to impurities. Butter-fat colored with any of these gave exactly the same coloration with hydrochloric acid alone as that shown by sesame-oil with hydrochloric acid and furfural; and although the author has no knowledge that these are employed to colour butter, there is nothing to exclude their use, whilst it is possible that other coloring matters may behave in a similar manner. C. A. M.

Estimation of Glycerol in Wines. F. Bordas and S. de Raczkowski. (*Ann. de Chim. Analyt.*, vol. ii. [20], pp. 381-383.)—The first step is to separate the glycerol by distillation in a current of steam. This is effected by heating 25 c.c. of the wine—previously neutralized by potassium hydroxide—in a short-necked 300 c.c. flask immersed in a brine-bath, the temperature of which is gradually raised to 110° C. The removal of the alcohol and more volatile substances is facilitated by connecting the apparatus with an exhaust; and as soon as these are all distilled, a current of steam is passed through the liquid in the flask (still kept at 110° C.) during three hours, when the glycerol distils over. At the close of the operation the action of the exhaust is continued until the delivery-tube is thoroughly cooled down. The distillate containing the glycerol is then made up to 200 c.c. for titration. When the wine is poor in glycerol, 50 c.c. should be taken for the experiment, in which case the steam is passed for an extra half-hour. A condenser at the rear of the receivers prevents any increase of pressure in the distilling flask.

The glycerol is estimated colorimetrically: four test-tubes are taken, into each of these 5 c.c. of the before-mentioned solution are placed along with *exactly* 2·5 c.c. of pure concentrated sulphuric acid, and 0·5, 1, 1·5, and 2 c.c. of a solution of potassium bichromate (24 grammes per litre) added to them respectively. The contents of one of the tubes will, after boiling, exhibit a yellowish-green tinge, and this one is selected as the basis for a second series of tests wherein the quantities of bichromate added are graduated by one-tenth of 1 c.c. above and below the quantity employed for the tube in question. From this series is selected the tube immediately preceding that one in which the liquid is of a slightly yellowish-green tint; and since 1 c.c. of the bichromate equals 0·0025 of glycerol, the volume of bichromate employed, multiplied by five, gives the quantity of glycerol per litre of wine. In case of doubt between two tubes, the mean is taken, but such instances are hardly likely to occur.

In very sweet wines the complete extraction of the glycerol is prevented by the opposition of the syrupy residue to the penetration of the steam. C. S.

On the Microscopical Examination of Food Substances. A. Hebebrand (*Forsch. Ber.*, 1897, iv., 306, 307.)—The author has found a preliminary treatment of the substance (suspended in an alkaline solution) with chlorine to give the most satisfactory results, both as regards the clearness of the structure and the quantitative estimation of foreign substances. In his experience, the reagents usually employed, such as aqua regia and caustic soda, act too energetically in many instances,

and cause the proportion of foreign substance to be different from what it was in the original sample. Or if, on the other hand, weaker reagents are used, the structure does not appear with sufficient clearness.

A small quantity of the finely-powdered substance is mixed with 10 to 15 c.c. of a solution of 7 grammes of dry sodium carbonate in 100 c.c., and chlorine passed in for a few minutes (from two to ten, according to the nature of the substance), care being taken that the reaction remains alkaline. The liquid is then diluted with water, and the bleached material separated, washed twice with water, and examined.

This method is claimed to give especially good results with starches, and in some cases has enabled the author to make a correct quantitative determination of rye meal in the presence of wheat meal owing to the former being attacked by the chlorine much more rapidly than the latter. The results, however, cannot always be relied upon. C. A. M.

Copaiba Balsam and Gurjun Balsam. L. F. Kebler. (*Amer. Jour. Pharm.*, 1897, lxix., 577-579.)—The different products described in the U.S. Pharmacopœia as balsam copaiba, oil of copaiba, mass copaiba, and resin copaiba, are derived from shrubs and trees of the genus Copaiba, all of which, with the exception of two African species, are natives of tropical America. According to J. C. Umney, the substances obtained from the latter differ very much from the more common American products.

The author has examined a large number of commercial samples, with the object of obtaining data as to their composition. In every case his modification of the glacial acetic acid test (*Amer. Jour. Pharm.*, lxvii., 394) was found reliable for the detection of Gurjun balsam. The samples in the subjoined table marked 'copaiba' are said to be representative of the commercial article. Of the Para samples, all are good with the exception of that with a specific gravity of 0·9874 at 15° C., which in the author's opinion is not a normal Para copaiba, but a more concentrated oleo-resin. The solidifiable copaiba may also be taken to fairly represent those found in commerce. The author has never met with a sample with a specific gravity below 0·9800 at 15° C., which solidified well:

Source.	Kind of Balsam.	Sp. gr. at 15° C.	Sp. gr. at 25° C.	Per cent. of Oil distilled from Metallic Bath.	Boiling-point of such Oil C°.	Sp. gr. of such Oil at 15° C.	Sp. gr. of Steam-distilled Oil at 15° C.	Sp. gr. of Steam-distilled Oil at 25° C.
Carthagena ...	Copaiba	0·9560	0·9506	53	250–265	0·9207	0·8997	0·8981
South America ...	,,	0·9416	0·9372	56	253–268	0·9174	0·9014	0·9000
Central America	,,	0·9526	0·9467	76	250–274	0·9231	0·9132	0·9067
Collected in 1846		0·9410	0·9381	62	253–270		0·9036	0·8978
South America ...	Para	0·9254	0·9200	90	258–270	0·9116	0·9079	0·9066
		0·9661	0·9583	88	254–268	0·9100	0·9093	0·9037
		0·9874	0·9818	54	253–265	0·9346	0·9019	0·9100
		0·9176	0·9116	92	256–268	0·9150	0·9951	0·9043
		0·9146	0·9101	90	254	—	0·8936	0·8904
	solidifiable	0·9926	1·000	23	260–269	0·9283	0·9201	0·9172
Commerce	Gurjun	0·9576	0·9516	—		—	0·9200	0·9146
,,	,,	0·9796	0·9722	54	245–263	0·9202	0·9192	0·9141
,,	,,	0·9531	0·9476	66	240–260	0·9146	0·9930	0·9176

The demand for solidifiable copaiba, mass copaiba, and resin copaiba, is very limited, and the article most in use is a copaiba containing 40 to 60 per cent. of oil. In the U.S. Pharmacopœia only the solidifiable balsam copaiba (properly an oleo-resin of copaiba) is recognised, and the author considers that the official requirements for it are inadequate. Thus he states that the residue is not necessarily brittle in the absence of any fixed oil, that the range of specific gravity is too low, and that a better limit would be 0·9800–1·0173. The test for Gurjun balsam, by heating the substance to 130° C., is said to be erroneous, for none of the samples examined congealed at that temperature, but only became slightly more viscous. He considers that more stringent tests are required for resin copaiba, since at present almost any resin will answer the requirements. C. A. M.

Detection of Turmeric in Powdered Rhubarb. A. Jaworowsky. (*Pharm. Zeits. Russl.*, 1897, xxxvi., 543; through *Chem. Zeit. Rep.*, 1897, 281.)—One gramme of the sample is shaken for some minutes with 10 c.c. of chloroform, the liquid filtered, diluted with fifteen times its volume of petroleum spirit, and divided into two portions. One is shaken with 2 or 3 c.c. of sulphuric acid and the other with 1 or 1·5 c.c. of a saturated solution of borax. If the rhubarb is pure, the chloroform has a straw-yellow colour, which disappears on dilution. On addition of sulphuric acid, the acid becomes brown, and the rest of the liquid remains colorless. The borax causes no change. In presence of turmeric, the chloroform is yellowish-brown, and exhibits a green fluorescence. Petroleum spirit yields a flocculent yellow precipitate, but neither the colour nor the fluorescence of the chloroform is affected by it. The sulphuric acid itself is colored a magenta red, which quickly alters to a red and then yellow-brown; the other liquid turns violet. The borax solution becomes violet, but the spirituous layer is unchanged in its appearance.

F. H. L.

The Important Constituents of Taraxacum Root. L. E. Sayre. (*Amer. Jour. Pharm.*, 1897, lxix., 543-546.)—The most important constituents identified by the author in taraxacum root are: (1) a resin, soluble in chloroform and ether, and insoluble in alcohol; (2) a resin soluble in alcohol; (3) *taraxacerin*, a white, wax-like substance; (4) a bitter principle, *taraxacin*, which in concentrated solution is precipitated by many alkaloidal reagents.

Attempts to isolate the bitter principle in crystalline form all proved unsuccessful, for it was found impossible to obtain crystals free from oleo-resinous drops, though acetone appeared to be the most promising solvent.

Taraxacerin, the wax-like body referred to above, separates from alcoholic solution in cauliflower-like forms. It melts at about 45° C., and has an empirical formula corresponding to $C_9H_{15}O$, or a multiple of this.

The amount of ash in one sample of the root dried at 100° C., was 11·13 per cent. with the following composition:

	Per cent.
Silica	43·27
Alumina	18·07
Ferric oxide	0·80
Calcium oxide	5·75
Magnesium oxide	6·60
Potassium oxide	13·83
Sulphuric acid (SO_4)	4·22
Phosphorus pentoxide	trace
Carbon dioxide	6·53
Chlorine	1·20
	100·27

C. A. M.

ORGANIC ANALYSIS.

Examination of Wax. G. Buchner. (*Zeit. für öffent. Chemie*, iii., 570.)—In the *Chem. Revue über die Fett und Harzindustrie*, 1897, p. 261, K. Dietrich published some results obtained on examining bees-wax by Hübl's method and by Henriques' cold saponification method, leading to the conclusion that the constants obtained by Hübl's method are not always to be depended upon. The author has examined two samples of bees-wax, of unquestionable purity, by both methods, (*a*) by dissolving in 25 c.c. of warm petroleum benzine in the ordinary way, whereby some loss of benzine by evaporation is sustained, and (*b*) by dissolving under special precautions, to avoid such loss. The following results were obtained:

HÜBL'S METHOD.

	I.		II.	
	(*a*)	(*b*)	(*a*)	(*b*)
Acid number	18·37	18·50	17·23	17·76
Ether number	75·98	75·81	75·98	74·60
Saponification number	94·35	94·31	93·21	92·36
Ratio	4·15	4·10	4·41	4·20

HENRIQUES' COLD SAPONIFICATION METHOD.

Petroleum benzine boiling at 100° to 150° C.

	I.		II.	
	(*a*)	(*b*)	(*a*)	(*b*)
Acid number	19·48	19·48	19·11	19·11
Ether number	72·81	72·81	72·81	72·81
Saponification number	92·29	92·29	91·92	91·92
Ratio	3·74	3·74	3·90	3·90

Petroleum benzine boiling at 50° to 80° C.

	I.		II.	
	(*a*)	(*b*)	(*a*)	(*b*)
Acid number	18·39	19·48	17·64	19·11
Ether number	53·60	71·09	54·53	68·16
Saponification number	72·99	90·57	72·27	87·27
Ratio	2·91	3·65	3·09	3·56

The lower the boiling-point of the benzine, the more difficult is it to effect the solution of the wax, and the more incomplete is the saponification. For cold saponification, petroleum benzine boiling at 100° to 150° C. is indispensable; but the results obtained are very exact, surpassing those yielded by Hübl's method. Hübl's method has, however, the advantage of rapidity of execution. H. H. B. S.

Examination of Rose-Oil. F. Dietze. (Reprint from *Südd. Apoth. Zeit.*, 1897, No. 89; through *Chem. Zeit. Rep.*, 1897, 288.)—The author states that geranium-oil can be detected in rose-oil by determination of the saponification number, and he has obtained the following values for this constant in different specimens of the two substances: Rose-oil from Germany, 8·6; Bulgaria, 9·2; Turkey, 8·9; geranium-oil from France, 59·1; Africa, 53·1; Spain, 75·3; India, 32·5. He also suggests the annexed specification: Genuine rose-oil should have a specific gravity not exceeding 0·870 at 15°; its solidifying point should not be lower than 15° or 20°; its saponification number not higher than 9·5 or 10; and its rotatory power in a 100 mm. tube at 20° C. not more than −1° 30′. F. H. L.

Properties of Some Essential Oils. Reports of Schimmel and Co. and H. Haensel. (*Deutsche Chem. Zeit.*, 1897, xii., 385.)—Oil of Bay.—When freed from terpenes this oil separates into two immiscible layers; the upper one is yellow and has a sp. gr. of 0·9533; the lower is a brown commercial product, sp. gr. 1·0358; and the terpenes themselves have a sp. gr. of 0·8146 (all at 18° C.).

Oil of Aniseed.—This is liable to be adulterated with oil of fennel and stearoptene.

Oil of Cananga is largely sophisticated with cocoa-butter, which raises the saponification number; but as other esters (Ylang-ylang oil), boiling at a lower temperature, are apt to be present, this test alone is not satisfactory. Its behaviour with alcohol and in a freezing mixture are, however, characteristic:

	Sp. gr.	Rot. Power.	Sap. No.	Solubility in Vols. of 95 per cent. Spirit.	Behaviour in Freezing Mixture.
Pure Cananga, crude	0·916	−21° 27′	17·8	0·5 to 2.	Liquid.
,, ,, refined	0·915	−20° 48′	16·0	,,	,,
Mixed with cocoa-fat	0·919	−17° 1′	83·7	Sep. of oil.	Becomes buttery.
Ditto and rectified...	0·908	−21° 50′	14·4	0·5 to 2.	Liquid.

Oil of Cardamoms. (1) From *Malabar*.—Sp. gr., 0·943; rot. power, +34° 52′ at 19°; sol. in 4 parts of 70 per cent. alcohol; sap. no., 137 (? 13·7). Contains acetic acid, cineol, and dextro-terpineol. (2) From *Siam*.—Sp. gr., 0·905; r. p., +38° 4′ at 42°; sol. in 1·2 parts of 80 per cent. alcohol; sap. no., 18·8; after acetylation, 77·2 (= 22·5 per cent. of borneol). Contains + borneol and camphor. (3) From *Cameroons*.—Sp. gr., 0·907; r. p., −20° 34′; sol. in 7·8 parts of 80 per cent. alcohol. Contains cineole. (4) *Oil of Grains of Paradise*.—Sp. gr., 0·894; r. p., −3° 58′; sol. in 10·11 parts of 90 per cent. spirit.

Oil of Cedar-wood.—Cedrene, the hydrocarbon of this oil, after distillation over sodium, boils at 131°-132° at 10 mm. pressure; its r. p. is −47° 54′; and it is a

sesquiterpene. With chromic and glacial acetic acids it yields a ketone (cedrone), boiling at 147° to 151° (7·5 mm.). Its oxime boils at 175° to 180° (8 mm.), and forms an acetate boiling at 185° to 190° (9 mm.). By reduction with sodium in ethereal solution the ketone gives liquid isocedrol (b.p., 148° to 151° at 7 mm.), the benzoic ester of which boils at 221° to 223° C. at 6 mm.

OIL OF CITRON.—For this and the two next oils, the Italian Government has specified the following (far too severe) requirements: Sp. gr. at 15°, 0·857 to 0·862; r. p. at 20°, between +57° and +67° in a 100 mm. tube. When 50 c.c. are fractionated, the first 5 c.c. distilled must have an opticity not exceeding 5° less than the original oil. (According to Soldiani and Bertè, when half the oil is distilled the distillate should have a r. p. higher than that of the residue, and also than that of the sample.)

OIL OF ORANGE.—Sp. gr., 0·847 to 0·853 at 15°; r. p., +96° to 98° at 20°.

OIL OF BERGAMOT.—Sp. gr., between 0·882 and 0·886 at 15°; r. p., between +8° and +20° at 30°; the ester content, determined on 2 or 4 grammes of the oil with normal potash in a flask fitted with inverted condenser, not below 30 per cent.; 5 grammes evaporated on the water-bath not to leave more than 6 per cent. of residue; must be soluble in 0·5 vol. of 90 v/v* alcohol, and remain clear on further dilution.

AMERICAN FIR-OIL (*Pinus sylvestris*).—Sp. gr., 0·884; r. p., −24° 8′ at 18°; sol. in 8 or more parts of 90 per cent. spirit with slight turbidity; sap. no., 34·8 (the ester is probably bornylic acetate; if so, it equals 12·1 per cent.). The English oil is also lævorotatory, while the German and Swedish specimens are dextrorotatory.

HEMLOCK-OIL (*Abies Canadensis*).—Sp. gr., 0·911; r. p., −25° 22′ at 16°. Contains 38 per cent. of lævorotatory bornylic acetate, also lævorotatory pinene, and is sol. in 1 or more parts of 90 per cent. alcohol.

SPRUCE-OIL (*Picea nigra* or *alba L.*).—Sp. gr., 0·913; r. p., −23° 50′ at 81°; sol. in 0·5 vol. of 90 per cent. alcohol, and yields with 5 vols. a strongly opalescent liquid. It also contains lævorotatory bornylic acetate and lævorotatory pinene, and cannot be distinguished either by odour or otherwise from the previous oil.

GUAIACUM-OIL in its normal state melts at 45° or 50°; but for the preparation of essences a more fluid sort is preferred, which remains liquid at summer temperatures. The crystalline alcohol, guaiacol, is odourless. The oil forms a favourite perfume, and is used in soap.

OIL OF MYRRH.—Bisabol myrrh, coming from the interior of Somaliland, has been investigated by Tucholka. On shaking out the alcoholic resin solution with petroleum spirit, evaporating the solvent and distilling the residue with steam, a pale-yellow limpid oil was obtained, having a characteristic odour, the yield being 7·8 per cent. Dried over calcium chloride, its sp. gr. at 24° was 0·8836; it boiled between 220° and 270°; and its optical values were:

$$n_{D\,22\cdot3}^{\circ} = 1\cdot4874, \text{ and } [\alpha]_{D\,24}^{\circ} = -14^{\circ}\ 20' \text{ (100 mm. tube).}$$

By the action of HCl gas on its ethereal solution, 65 per cent. of a hydrochloride

* Per cent. by volume (*cf.* ANALYST, xxii., 89).

was produced, which, after repeated crystallizations from alcohol and ether, melted at 79·3°. Sodium acetate and glacial acetic acid split this up into a hydrocarbon with sp. gr., 0·8914 at 17°; b. p., 259° to 260·5°; and refractive index, 1·4608. It appears to be a new terpene, and has been named bisabolene.

Oil of Petit-grain.—Passy has lately found that this oil contains considerable quantities of geraniol, partly free and partly as the acetic ester; these seem to exert much influence on its odour.

Oil of Peppermint.—To estimate the menthol, 5 grammes are heated under an inverted condenser with (exactly) 5 c.c. of acetic anhydride for half an hour, and, after cooling, the excess is titrated with normal soda, the strength of the anhydride being determined in the meantime in a similar manner. The volume of alkali neutralized × 0·156 gives the menthol.

Oil of Rosemary has been proved to contain pinene. It should answer to the following requirements: sp. gr., above 0·9; r. p., +; sol. in 0·5 vol. of 90 per cent., and also in 10 vols. of 80 per cent. alcohol; on distillation, the first 10 per cent. must have a + r. p.

Oil of Sandal-wood should be examined by acetylation, determination of sp. gr., r. p., and sol. in 70 per cent. spirit. Colour reactions are useless.

Oil of Vetiver (*Cuscus*) differs largely in its constants. When pure it has sp. gr. 1·019 to 1·027 at 15°; r. p., +25° or 26°; sap. no., 70 to 80; sol. in 80 per cent. alcohol 1·5 to 2 vols.

Oil of Xanthorrhœa.—On distilling the resin from the Australian "yellow grass tree," 0·37 per cent. of yellow oil smelling like storax is obtained. Sp. gr., 0·937; r. p., −3° 14′; acid no., 4·9; sap. no., 74·3; ester no., 69·4. The free acid is cinnamic, and melts at 133°. On saponification, 20 per cent. of the same substance may be recovered.

Oil of Marvel of Peru (*Tanacetum balsamita L.*).—Prepared from the flowers; yield, 0·064 per cent.; odour like tansy; sp. gr., 0·943; r. p., −53° 48′ at 16°; sap. no., 21. Insol. in 80 per cent., but sol. in 1 or 2 vols. of 90 per cent. alcohol, further additions causing turbidity and separation of white flocks. Distils between 207° and 283°.

Heliotropin.—A product having this name has been put on the market by a Swiss firm. It consists of heliotropin, 90 per cent.; vanillin, 10 per cent.; solution of ionone, 0·5 per cent.; and a trace of rose-oil.

Ionone.—A crystalline preparation called "ionone" has been met with, composed of one of the artificial musks, which had been given a violet-like odour by being crystallized from ionone. It contained nitrogen in the form of a nitro compound, exploded on heating with sodium, while its odour changed from that of violets to that of musk on boiling with water.

Concrete Violet.—This is an American product belonging to the class of fixed scented oils. It consists of a mixture of fat, oil of iris (sweet flag) and artificial musk. The latter crystallizes out of its solution in petroleum ether. If the molten substance is shaken with 80 per cent. alcohol, the fat filtered off, and the solution treated with parabromphenylhydrazine, a yellow precipitate is produced, which

crystallizes in transparent scales from hot spirit (m. p., 156°). The odour is therefore due to irone, and not ionone.

Myristic acid is much used as the fat or solid matter in these fixed scented oils; and the following figures give approximately the amount of it contained in different varieties: Jasmine, 50 per cent.; elder flowers, 49 per cent.; acacia, 69 per cent.; fixed rose-oil, 73·8 per cent.; reseda (mignonette), 85 per cent.; violet flowers, 86 per cent.

[NOTE BY ABSTRACTOR.—This article also contains a number of further details respecting these oils, etc.; but they do not appear to possess any analytical interest.]

F. H. L.

Application of Maumené's Method to the Examination of Essential Oils. M. Duyk. (*Rev. de Chim. Analyt. Appl.*, vol. v., pp. 442-445.)—In order to prevent the violence of the reaction which ensues, in applying this test to essential oils, it was found necessary to dilute the oil with an inert substance, and for this purpose commercial, colorless, liquid paraffin was employed.

To apply the test, 4 c.c. of the liquid paraffin and 1 c.c. of essential oil are mixed by shaking in a 15 c.c. flask fitted with a thermometer, the bulb of which is immersed in the liquid. Two c.c. of pure monohydric sulphuric acid are then carefully pipetted down the side of the flask, and, after the thermometer has been read, are shaken up with the other contents, the resulting increase of temperature being read off as soon as the maximum is obtained. The difference gives the increase occurring under the conditions of the experiment.

The subjoined figures were obtained by the foregoing method, the initial temperature being 120° C., and the weight of the flask 30 grammes:

TERPENE HYDROCARBONS.

Cymene, $C_{10}H_{14}$, from turpentine essence, 4° C.
Pinene, $C_{10}H_{16}$, from turpentine essence, 36° C.
Limonene, $C_{10}H_{16}$, from citron essence, 26° C.
Carvene (Limonene), from caraway essence, 26° C.
Thymene, $C_{10}H_{16}$, from thyme essence, 13° C.
Cedrene, $C_{15}H_{24}$, from cedar essence, 15° C.

OXYGENATED COMPOUNDS.

Anisol, from anise essence, 22° C.
Thymol, from thyme essence, 7° C.
Safrol, from sassafras essence, 33° C.
Carvacrol, from thyme essence, 4·5° C.
Apiol camphor, from parsley essence, 32° C.
Eugenol, from clove essence, 27° C.
Geraniol, from geranium essence, 31·5° C.
Menthol, from mint essence, 9° C.
Linalool, from linaloe essence, 38° C.
Eucalyptol, from eucalyptus essence, 22° C.
Terpinol, from turpentine essence, 26° C.
Santalol, from santol essence, 33° C.

Citral, from lemon-grass essence, 40° C.
Anisic aldehyde, 14° C.
Benzaldehyde, from bitter almond essence, 9° C.
Menthone, 11° C.
Citronellol, from citronella essence, 33° C.
Carvol, from caraway essence, 30° C.

ESSENTIAL OILS.

Santal—*East Indian:* Samples from different makers ranged between 22·5° C. and 31° C. That prepared by the author gave 30° C. *West Indian:* 12°, 16°, 18° C., according to the maker.
Cedar: different makes, 16° to 18° C.
Copaiva: 24° C. Gurjum: 21° C.
Mint: Mitcham, 16° C.; Saxon, 13° C.; Crépue Japanese, 20° C.
Lavender: French, 37°, 36°, 34° C.; English, 33° C.
Spike lavender: 29·5° to 30° C.
Rose: Pure Turkish, 34·5° C.
Geranium: Turkish, 24° C.; freed from hydrocarbons, 25° C.
Citron: 25° to 26·5° C.; freed from hydrocarbons, 29° C.
Portugal orange: 37° C.; Neroli, 36·5° C.; Petitgrain, 33·5° C.
Red thyme: 23·5° C. Rectified thyme: 22·5° C. Eucalyptus: 24° C.
Clove: 28° C. Bitter almonds: 9·5° C.
Canella: Ceylon, 20° C.; China, 14° C.
Caraway: French, 28° C.; German, 30° C.; Cumin, 17° C.
Green anise: 17° C. Aniseed (badiane): 21° C. Fennel: 19·5° C. C. S.

Preparation of Sugar Liquors for Polarimetry. J. Diamant. (*Chem. Zeit.*, 1897, xxi., 981.)—The author recommends the use of zinc-dust to remove the excess of lead from sugar solutions which have been decolorized by means of lead acetate. The precipitates settle quickly, the liquids are much clearer than before treatment with zinc, and the salts of the latter metal are without influence on the readings of the polarimeter. F. H. L.

Use of Alcohol for the Reduction of Cuprous Oxide in Sugar Determinations. G. Bruhns. (*Zeit. für. öffent. Chemie*, iii., 573.)—The cuprous oxide obtained in gravimetric sugar-determinations by Fehling's solution is reduced by means of alcohol, and the copper weighed in the metallic state. The procedure is as follows: The copper precipitate is filtered off and ignited in a platinum crucible. The crucible is then covered with a lid, having a hole in the centre and 1 c.c. of methyl alcohol added, a drop at a time, through the hole, the crucible being meanwhile maintained at a red-heat. The lamp is now turned out, and 1 c.c. of methyl alcohol run in at one time. The drops of alcohol at first added assume the spheroidal state, and consequently evaporate but slowly, so that the vapour has time to exercise its reducing action upon the oxide. The 1 c.c. added after the lamp has been turned out prevents, by its evaporation, the entry of air into the crucible, and thus protects the copper, while hot, from oxidation. The vapour of the alcohol is finally ignited,

and when it has burnt out, the crucible lid is removed and the crucible exposed to the air until no further odour of alcohol or formaldehyde is given off. It is allowed to remain five minutes in the balance-case, and is then weighed. The weight is quite constant, but, for the sake of control, the crucible can be heated again for a short time and re-weighed.

Ethyl alcohol of 99 per cent. can be substituted for methyl alcohol; but in this case a little carbon separates, which discolors the copper, though scarcely any increase in weight is perceptible. H. H. B. S.

Reactions of the Glucotannins of the Cinnamic Acid Series. H. Kunz-Krause. (*Chem. Zeit.*, 1897, xxi., 941.)—These bodies exhibit several reactions common to the whole group, among which the following are the most important. Dissolved in alcohol and treated with metallic sodium, they give a yellow precipitate, which is easily soluble in water. They form yellow lead-salts. They evolve hydrocyanic acid with Liebermann's reagent in the cold. They yield glucosazones on the addition of phenylhydrazine.

The best-known member of the series is caffetannic acid (glucosyldihydroxycinnamic acid), which is identical with matétannic acid. This substance readily absorbs water, alcohol, and ammonia. It combines with orthophenylenediamine; and its osazone melts at 180° C. With uranium acetate it yields a reddish-brown precipitate, but no carbon dioxide is given off when the products of the reaction are insolated. With chloral hydrate it forms a yellow, rather unstable compound. By itself, and in presence of acids, caffetannic acid is quite permanent; even on warming alone or with dilute acids it is not easily attacked; but on heating with strong hydrochloric acid it is converted into an amorphous, humus-like body. Bromine water splits up the molecule into a soluble sugar and a brown insoluble substance containing the halogen. The reaction, however, does not proceed smoothly, so that it is impossible to estimate caffetannic acid by determination of the sugar with Fehling's solution. Nevertheless, the decomposition proves that these glucotannins are true glucosides, a point about which there has previously been some uncertainty.

F. H. L.

Volumetric Estimation of Uric Acid. J. W. Tunnicliffe and O. Rosenheim. (*Centralbl. Physiol.*, 1897, xi., 434; through *Chem. Zeit. Rep.*, 1897, 280.)—This process depends on the fact that piperidine combines with uric acid in molecular proportions (4·25 grammes of base equal 8·4 grammes of acid) to form a soluble salt. A $\frac{N}{20}$ solution of the former is prepared by dissolving about 4·2 grammes of piperidine in 1 litre of water, standardizing it on hydrochloric acid of equivalent strength, phenolphthalein being used as indicator. The sample of uric acid is suspended in water, heated nearly to the boiling-point, and the reagent run in; neutrality being shown either by the liquid becoming clear or by the use of phenolphthalein as before. Although the solubility of the urate at 15° is 5·3 per cent., it is better to employ hot solutions; and there is no danger of losing any piperidine by volatilization, as the reaction is instantaneous. The method is particularly adapted for the examination of urine; and the uric acid is best obtained in a form suitable

for titration by Hopkin's process. This consists in separating the acid as its acid ammonium salt, and then decomposing the latter with HCl. The crystals must, of course, be washed on a filter till free from hydrochloric acid; but 15 or 20 c.c. of water are usually sufficient for the purpose. The paper is then perforated, and the uric acid is rinsed into a flask with 20 or 30 c.c. of hot water. F. H. L.

INORGANIC ANALYSIS.

The Determination of Lead in Lead Ores. R. K. Meade. (*Jour. Amer. Chem. Soc.*, 1897, xix., 374-377.)—The following is recommended as a simple and rapid method for the determination of lead in galena and lead ores in general in the absence of barium, strontium, and calcium, and of metals oxidized to insoluble compounds by nitric acid: One gramme of the ore, or more if poor, is heated in a covered platinum dish on a sand-bath with 40 to 50 c.c. of a mixture of one part of sulphuric acid (specific gravity 1·84) and three parts of nitric acid (specific gravity 1·42) until the action has apparently ceased. 10 to 15 c.c. of hydrofluoric acid are then added, and the solution evaporated until fumes of sulphuric acid begin to come off. The dish is cooled, and after dilution of the liquid to about 100 c.c., the insoluble matter is filtered off, washed first with a 2 per cent. solution of sulphuric acid and then with alcohol, dried, ignited apart from the filter-paper, and weighed as lead sulphate.

Experiments with a solution of lead nitrate proved that no lead was lost by volatilization as fluoride, although the results were slightly lower on evaporating 25 c.c. of the solution with sulphuric and hydrofluoric acids than with sulphuric acid alone, the averages of six determinations being 0·4280 gramme and 0·4297 gramme respectively. 25 c.c. of the solution, to which 0·2 gramme of silica had been added, when evaporated with the two acids, gave 0·4285 gramme of lead sulphate.

The analysis of different lead ores by the method given by Fresenius (*Quant. Anal.*, 213), and by this method, after calcium, barium, and strontium had been proved to be absent, gave the following results:

	Fresenius. Per cent. lead.	New Method. Per cent. lead.
Galenite	77·34	77·46
Pyromorphite	75·26	75·35
Cerussite	76·46	76·53

Barium and strontium are rarely found associated with lead, and calcium, when present as calcite, may be removed by treating the ore with hydrochloric acid and filtering. The residue containing the lead is then treated with sulphuric, nitric, and hydrofluoric acids, as described above. C. A. M.

Note on the Analysis of Bronze and Brass. F. Fatet. (*Ann. de Chim. Analyt.*, vol. ii. [20], pp. 383-385.)—Having found that the titration of zinc in an ammoniacal solution containing copper cannot be accurately performed by means of sodium sulphide after the copper has been decolorized by potassium cyanide, and also that the presence of zinc falsifies the titration of the copper by the last-named reagent, the author examined the method proposed by F. Jean for estimating these

metals in impure galena—viz., titration of the two metals by sodium sulphide in an ammoniacal solution, then acidifying with hydrochloric acid, to redissolve the zinc, and, after boiling the filtered liquid and adding ammonia, titrating the latter metal with the same reagent, the copper being estimated from the difference between the two tests. He finds that this method affords an accurate and speedy means for the analysis of bronze and brass.

To check the end point of the precipitation of the sulphides, he recommends the test proposed by Mohr—*i.e.*, the brown arc formed at the point of contact between a drop of an alkaline solution of lead and a drop of the test liquid placed on filter-paper. C. S.

Estimation of Iron with Permanganate in Presence of Hydrochloric Acid. M. Hauffe. (*Chem. Zeit.*, 1897, xxi., 894.)—The author employs the following combination of well-known methods: *Reagents necessary.*—(1) Permanganate solution containing 3 grammes per litre, standardized on pure iron under the conditions as detailed below; (2) 100 grammes of crystallized manganese sulphate, dissolved in 1,300 c.c. of water and 200 c.c. of pure concentrated sulphuric acid; (3) a cold-saturated solution of mercuric chloride; (4) a dilute solution of stannous chloride. *Process.*—A sufficient quantity of the material under examination to serve for several titrations is dissolved in a moderate excess of warm HCl, and, if organic matter is present, the liquid is treated three times with some strong permanganate, and boiled till free from chlorine, any oxide of manganese thrown out being taken up in a few drops of acid. When cold, it is diluted to a definite volume, and an aliquot portion (50 to 200 c.c.) is heated to boiling in a beaker capable of holding 2 litres. Stannous chloride is then run in from a burette till, after all the iron is reduced to the ferrous state, enough of the reagent still remains unoxidized to give a slight precipitate when 100 c.c. of the mercuric chloride solution are next added (*cf.* the somewhat similar process, ANALYST, xxii., 299). If the excess of stannous chloride is too large, the precipitate of mercurous chloride becomes so bulky as to hide the pink colour in the final titration, but if the mercuric chloride does not produce any precipitate, too little tin has been employed. Finally, 60 to 100 c.c. of the manganese sulphate are introduced, the liquid cooled as rapidly as possible, 1 litre of water added, and the whole titrated with the permanganate. When only trifling amounts of iron have to be determined, a blank experiment should be carried out on 10 c.c. of the stannous chloride, 10 c.c. of HCl, and 50 c.c. of water, warmed together, and treated with $HgCl_2$, etc., as above, in order to make proper allowance for the few drops of permanganate required to yield a visible pink in such a large mass of fluid (1·5 litres).

Iron ores should be dried at 105°C., and 5 grammes dissolved, first by boiling with HCl (specific gravity 1·19), and then by fusion with alkali carbonates in the ordinary manner; and if the proportion of silica is not so high as to cause the liquids to boil badly, it need not be removed. The volumes employed should be adjusted to allow either 0·5 or 1·0 gramme of the mineral to be titrated according to its richness in iron. In the case of phosphates, 25 grammes should be dissolved in 50 or 60 c.c. of strong acid, and 5 grammes taken for each analysis; but the amount of manganese

sulphate should be increased to 100 or 150 c.c., so that there may be no odour of chlorine perceptible during the addition of the permanganate. F. H. L.

Estimation of Alumina and Iron in Phosphates. F. Jean. (*Ann. de Chim. Analyt.*, vol. ii. [20], p. 403.)—The solution of the phosphate is made up to 100 c.c., and, after the phosphoric acid has been thrown down by the ordinary citro-magnesia method, 50 c.c. of the clear liquid are pipetted into a bulb flask, and there concentrated to 8 or 10 c.c. This quantity is boiled with 25 c.c. of 66 per cent. sulphuric acid and 5 c.c. of fuming nitric acid until colorless, the elimination of the organic matter being accelerated by the addition, at intervals, of a few crystals of sodium nitrate or a little nitric acid. When cooled, the liquid is diluted and treated with alcohol to precipitate calcium sulphate. After filtration the alcohol is boiled off; and, after the addition of a few crystals of potassium chlorate, the iron and alumina are thrown down by ammonia.

When manganese is present, the precipitate is fused in a platinum crucible with an alkali carbonate and potassium nitrate, the mass being taken up with water and the residue (iron oxide) weighed. Manganese is determined by titration, and the alumina estimated by difference. C. S.

The Behaviour of Thoria with Oxalic Acid and Ammonium Oxalate, and the Estimation of Thoria. C. Glaser. (*Zeit. anal. Chem.*, 1897, xxxvi., 213-219.)—In a former communication (*Chem. Zeit.*, lxiii., 612; ANALYST, xxi., 274-277) the author stated that the thoria recovered after the ammonium acetate treatment, when redissolved, could no longer be completely precipitated by adding ammonium oxalate, boiling, and allowing the liquid to cool. This he now finds to be due to the fact that thorium oxalate in nearly neutral solution is soluble in a large excess of ammonium oxalate, a statement which confirms the results of other observers. When there is not an excess of ammonium oxalate, no precipitation occurs in a boiling nearly neutral mineral acid solution of thoria, but on cooling the thorium oxalate comes down in crystalline form. Ammonium acetate only increases the solvent action of the liquid. By adding oxalic acid, or by making the solution strongly acid with hydrochloric acid, the thorium is precipitated quantitatively even when there is a large excess of ammonium oxalate.

The composition of the precipitate varies considerably, according to the method by which it is obtained. Thus, on adding oxalic acid to a solution of thoria before boiling, a precipitate of the normal oxalate is formed on cooling; but if the solution be boiled before the oxalic acid is added, the precipitate consists of a mixture of thorium oxide and oxalate, the oxide being sometimes partially precipitated before the oxalic acid is added.

On precipitating thorium by ammonium oxalate (not in excess) from a nearly neutral solution, the normal oxalate is not obtained, nor is it when the thorium is precipitated by ammonium oxalate from a solution made strongly acid with hydrochloric acid.

From these experiments it follows that a complete separation of thoria and

zirconia by means of ammonium oxalate is only possible when care is taken to avoid a greater excess of ammonium oxalate than is required for the solution of the zirconium oxalate, but when cerium is also present, it is very difficult to avoid an excess on account of the insolubility of cerium oxalate.

The author finds further that in separating cerium and thorium oxalates by means of ammonium acetate, a large excess of the latter must be avoided, since cerium oxalate is slightly soluble in ammonium acetate. Only a few c.c. of the reagent are required to dissolve out the whole of the thorium from the mixed oxalates.

As it was possible that these facts might make an alteration necessary in the scheme of analysis given in the previous paper, a sample of monazite sand was analysed by that method, but it was found that, notwithstanding its solubility in excess of ammonium oxalate, the whole of the thoria had separated out completely.

It is, however, advisable to examine the filtrate from the mixed oxalates of thorium and cerium, by adding ammonia in excess, filtering, dissolving the precipitate in hydrochloric acid in excess, adding oxalic acid, and leaving the liquid in a warm place.

When a determination of cerium is not required, the solution of the earths in sulphuric acid can be treated at once with an excess of hydrochloric acid and oxalic acid, and the precipitated thorium and cerium oxalates separated in the usual way by means of ammonium acetate.

The following remarks refer to the table of the more important reactions of the rare earths in the former paper:

1. The statement that thoria and ceria are precipitated by ammonium carbonate as well as by ammonia (Graham-Otto, part 2, 902-904) is incorrect.

2. Ammonium oxalate precipitates thoria in cold, but not in boiling, solution. An excess of hydrochloric acid precipitates the oxalate quantitatively on cooling.

3. The statement that thorium oxalate kept in solution by means of ammonium acetate and ammonium oxalate is partially precipitated by hydrochloric acid should read: that in the presence of a sufficient excess of hydrochloric acid the precipitation is complete.

4. Mention should be made of the solubility of zirconium oxalate in hydrochloric acid.

5. Cerium oxalate is slightly soluble in ammonium acetate. C. A. M.

Analysis of Sodium Bicarbonate. G. Lunge. (*Zeit. angew. Chem.*, 1897, 169-171).—This method, which was devised by Sundstrom, depends on the fact that a solution of sodium bicarbonate, when treated with sodium hydrate, is converted into carbonate. The end of the reaction is indicated by the excess of sodium hydrate giving a brown colour with silver nitrate solution, used as an outside indicator. From the result thus obtained, together with that of the total acidity determined by titration with normal acid, the composition of the sample can be calculated.

The solution of sodium hydrate is prepared by dissolving commercially pure caustic soda in water till the specific gravity reaches 1·16, precipitating with barium chloride, adding barium hydrate to saturation and diluting the solution to normal strength.

The use of ordinary normal sodium hydrate did not give satisfactory results, and, as the author points out, the barium hydrate in Sundstrom's solution at once detects the presence of any carbonate. C. A. M.

Analysis of Commercial Liquid Ammonia. A Lange and J. Hertz. (*Zeit. angew. Chem.*, 1897, 224-228.)—The authors find that the residue left on the evaporation of German liquid ammonia consists chiefly of water, pyridine bases, acetonitrile, ethyl alcohol, and a little machine oil; with ammonium carbonate, benzene, and naphthalene as occasional impurities.

For the valuation of liquid ammonia in practice, it is sufficient to determine the amount of residue without taking into account its composition. This is done by placing the bottle in a horizontal position, and screwing on to the valves an open steel tube. By opening the valve, from 20 to 50 grammes are allowed to flow into a previously-weighed Erlenmeyer flask, and rapidly weighed after the insertion of the stopper. This has two openings, through one of which passes a glass tube (*a*), bent at right angles, whilst the other communicates with a potash tube (*b*). The glass tube is kept closed, so that the evaporating ammonia must pass through the potash tube. The evaporation, which is complete in from two to three hours, may be assisted by an occasional shake. The residue which it still contains is warmed to (at most) 40° C., and a current of dried air passed into the flask through the glass tube (*a*) Both tubes are then closed, the apparatus again weighed, and the amount of residue calculated.

It was proved by experiments that the different samples taken were homogeneous in character, and that none of the impurities referred to were lost during the evaporation of the ammonia. Thus, on taking samples of 33 grammes each from the same bottle in one-quarter, in one, and in five minutes, the amount of residue obtained was 0·9, 0·9, and 1·0 per cent. respectively.

The apparatus used by the authors for the volumetric determination of the residue consists of a glass tube of from 30 to 40 mm. internal diameter, which is fused at the bottom to a narrow tube of about 5 mm. diameter, this part holding at least 1·1 c.c. The larger part of the tube contains about 100 c.c., and has a mark at 49 c.c. corresponding to 33·3 grammes of ammonia. The lower narrow portion of 1·1 c.c. is graduated into fifteen parts, so that each corresponds to 0·2 per cent. The tube is filled to the mark with ammonia, a grooved cork inserted, and the liquid allowed to evaporate spontaneously. This takes about three hours, but the operation may be completed in half an hour by placing the tube in water. The presence of much water in the residue is a source of error, since the latter may then retain about 25 per cent. of ammonia, but with the average constituents of the residue the error is trifling. C. A. M.

Separation of Bromine from a Mixture of Soluble Bromides and Chlorides. Baubigny and Rivals. (*Comptes Rendus*, October 11, 1897, p. 527; through *Ann. de Chim. Analyt.*, vol. ii. [22], p. 425).—The mixture is treated with copper sulphate and potassium permanganate, a current of air being passed through the liquid to remove

the bromine, which is absorbed by a 3 to 4 per cent. solution of soda contained in a tube fitted with glass balls, which are kept apart by constrictions in the tube.

The mixed salts and the copper sulphate are placed in a long-necked flask containing water, and after solution has been effected by the aid of heat and the liquid has cooled down, the permanganate is added, the flask is connected with the air-supply and the absorption-tube, and is heated on the water-bath. To prevent loss by evaporation, the air is saturated with moisture by being passed through water in a flask on the same water-bath.

When all the bromine is driven over, the cold alkaline liquid in the condenser-tube is decanted and treated with a little gaseous sulphur dioxide, followed by a 10 per cent. solution of silver nitrate and a fairly large amount of nitric acid. In the acid liquid the sulphurous acid reduces any oxygenated bromine products present, and if heat be then applied, the nitric acid oxidizes the residual sulphur dioxide and the silver sulphite formed, leaving only silver bromide behind as the insoluble residue.

The silver nitrate should be added along with, and not *after*, the nitric acid, to prevent loss of bromine. C. S.

The Detection of Traces of Bromine by means of Fluorescein. H Baubigny. (*Compt. Rend.*, 1897, cxxv., 654-657.)—The method here described is based on the fact that bromine converts fluorescein into its tetra-bromo derivative, eosin. Test-paper is prepared by steeping paper in a solution of pure fluorescein (40 to 50 per cent.) in acetic acid and allowing it to dry. When moistened and brought into contact with bromine, such paper assumes a rose coloration. To detect bromides (in the absence of chlorides) the bromine is liberated by warming the solution with copper sulphate 16 grammes, and potassium permanganate 0·7 gramme, in 100 c.c., and passing a slow current of air through the flask while the test-paper is applied to the delivery tube. The formation of eosin on the paper after a few minutes shows the presence of bromine. The test is not suitable for the detection of bromine in the presence of free chlorine, especially if the latter be present in large excess, since chlorine gives a white derivative with fluorescein which masks the effect of the bromine. In such cases the free halogens may be converted into chloride and bromide, and then tested by treating the solution with copper sulphate and permanganate without warming and passing air through the flask. When chlorine is present alone, the paper remains unchanged after ten minutes; but if as little as 0·001 gramme of bromide be present with 5 to 10 grammes of a chloride, rose-coloured streaks appear within four or five minutes. The presence of an iodide does not interfere with the test if it is first transformed into iodate by warming the solution with a little permanganate, for iodine is not then liberated on the subsequent addition of the copper sulphate. The author is still studying the method of proceeding in the case of insoluble bromides. The test-paper is said to keep as well as turmeric-paper. C. A. M.

On the Estimation of Sulphuric Acid as Ammonium Sulphate. F. Marboutin and A. Pecoul. (*Bull. Soc. Chim.*, 1897, xvii., 880, 881.)—Weinig described a method

for determining sulphuric acid in dilute solutions by adding an excess of ammonia, evaporating and drying the ammonium sulphate at 115°. Shiver (*Chem. Soc.*, 1895, 351) found that the results thus obtained were too low, and recommended the addition to the residue of a little strong ammonia, evaporation on the water-bath and drying at 120° C. The authors tested this modification on $\frac{N}{20}$ solutions, but their results were still too low. In order to determine under what conditions this source of error might be avoided, they evaporated 100 c.c. of the solution (yielding theoretically 343·6 milligrammes of ammonium sulphate) with an excess of ammonia at 65°, and brought the residue gradually up to 120°, with the following results :

	Milligrammes.
At 65° after forty-eight hours (the time required for evaporation)	343·5
The residue heated at 70° for twenty hours	343·5
75° „ „	343·7
80°	343·6
85°	344·2
90°	345·4
95°	346·2
100°	345·2
110°	344·8
120°	343·3

The authors attribute the increase of weight which takes place at 85° to the liberation of free sulphuric acid which absorbs moisture from the atmosphere.

C. A. M.

Note on the Separation of Silicic and Tungstic Acids. J. S. de Benneville. (*Jour. Amer. Chem. Soc.*, 1897, xix., 377-379.)—A common method of determining silicon in tungsten steel and ferro-tungsten is to dissolve the tungstic oxide on the filter by means of hot dilute ammonia, and so obtain the silica at once for determination. It has been pointed out by various chemists that precipitated silicic acid, and even amorphous silica, are perceptibly soluble in hot ammonia, and the author finds that this is also the case with cold ammonia. He therefore advocates the method, recommended by Arnold, of weighing the total oxides and volatilizing the silica with hydrofluoric acid, with the addition of a few drops of sulphuric acid, as the most satisfactory.

A sample of wolframite, containing 65·5 per cent. of tungsten trioxide, and by the volatilization method 5·61 per cent. of silica, gave, on dissolving the tungsten oxide from the silica by ammonia : (1) Insoluble silica, 3·84 per cent., and soluble silica, 1·64 per cent. ; and (2) insoluble silica, 3·22 per cent., and soluble silica, 2·20 per cent. The silica was mainly derived from intermixed mica. C. A. M.

The Determination of Silica in Blast Furnace Slag. G. H. Meeker. (*Jour. Amer. Chem. Soc.*, 1897, xix., 370-374.)—For the method here described there is claimed (1) greater accuracy than in the usual methods on account of more complete dehydration of silicic acid and thorough decomposition of the slag, and (2) greater rapidity and ease of execution.

Half a gramme of the finely-powdered slag is mixed in a basin with about 3 c.c.

of water, and then stirred up with 10 c.c. of concentrated hydrochloric acid, till the slag has dissolved as completely as possible. 40 c.c. of sulphuric acid (1 : 4) are then poured into the basin covered with an inverted funnel, and the liquid boiled until fumes of sulphur trioxide appear. After cooling, 10 c.c. of hydrochloric acid are added, the liquid diluted and boiled for one minute, the basin being covered with a watch-glass. The residue is then filtered off, washed five times with hot hydrochloric acid (specific gravity 1·10) and five times with hot water, ignited, and weighed. The silica is remarkably pure. In "chilled" slags the impurities amount to from 0·05 to 0·20 per cent.

This method has been compared with a method consisting of two evaporations to dryness with hydrochloric acid, the heat being regulated so as not to exceed 120° C., and finally purifying the silica with hydrofluoric acid, and has, as a rule, given results in close agreement, though somewhat lower (about 0·1 per cent.), owing to the more complete dehydration of the silicic acid. The only exceptions to such agreement were in cases such as slags associated with spinel, in which the treatment with hydrofluoric and sulphuric acids changed the impurities (*e.g.*, alkalies) in the spinel into other compounds, which would influence the final result.

The percentage of silica obtained by the ordinary method and by this method in a slag containing spinel residue were:

	Ordinary Method.	New Method.
Silica and impurities	35·42 ...	31·20
Impurities determined by treatment with hydrofluoric and sulphuric acids ...	5·88 ...	0·20
	29·54	31·00

Attempts to readily determine alumina and lime in the filtrate from the silica all failed, the results being much too low. C. A. M.

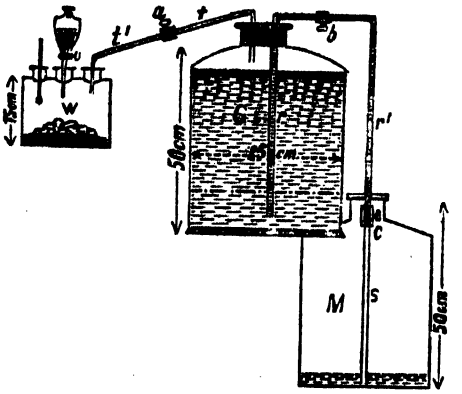
On the Estimation of Phosphoric Acid as Phosphomolybdic Anhydride. H. Pellet. (*Rev. Chim. Analyt. Appl.*, vol. v. [20], p. 405.)—The double precipitation indicated by Woy (ANALYST, xxii., 250) for obtaining a pure precipitate of ammonium phosphomolybdate is considered unnecessary. It is found that when a sufficiency of ammonium nitrate is used, the molybdic acid is not precipitated, and the dried precipitate of ammonium phosphomolybdate corresponds to 3·75 grammes of phosphoric acid or to 3·95 of phosphomolybdic acid.

The estimation can therefore be accelerated by substituting ignition for desiccation, provided the heat is applied slowly and the heating is not continued too long, otherwise the results may be too low. C. S.

Valuation of Calcium Carbide by Measurement of the Acetylene it evolves. F. Fuchs and F. Schiff. (*Chem. Zeit.*, 1897, xxi., 875.)—When olive-oil is shaken for half an hour with acetylene, it dissolves 48 per cent. by volume of the gas; but if a layer of the oil previously saturated and 15 mm. thick is floated on the surface of water contained in the holder of the apparatus described below, the further loss

of acetylene in the time taken by each experiment does not exceed 1·5 or 2·0 per cent.

The generator, as seen in the accompanying cut, consists of a three-necked Woulffe's bottle (*W*), carrying a thermometer, a stoppered funnel, and a leading tube for the gas, the bottom of the vessel being covered with asbestos to avoid danger of breakage by the heat produced during the decomposition of the carbide. The gas-holder (*G*) is a glass bottle 50 cm. high and 25 cm. in diameter, having a capacity of 15 litres, and filled with water bearing the film of oil already referred to. The inlet tube is cut off just below the cork, so that the gas does not come in contact with the water itself. The outlet for the displaced liquid is a tube passing through the same cork, reaching to the bottom of the vessel, bent twice at right angles, and having both legs of equal length. At its external extremity it is connected by means of a short piece of rubber tube provided with a screw clip to another length of glass tube, passing to the bottom of a second vessel (*M*) the same size and shape as *G*, which at the beginning of the test contains exactly 1 litre of water, and stands at a lower level. By this arrangement, when the tube between *G* and *M* is full of water, it acts as a syphon, so that the gas in *G* and also in the generator is under diminished pressure, and there is no danger that the water in the funnel of the latter will be blown out when the cock is opened.



About 30 grammes of carbide are taken for analysis, and 200 c.c. of water are used to decompose it, the rate of admission through the funnel being regulated with the stop-cock. Although the introduction of the water alters the capacity of the apparatus, if the generator is well shaken after the production of gas has ceased no error will arise, because water dissolves exactly its own volume of acetylene. In about an hour and a half the temperature of the generator will have fallen to its original point. The generator *W* is well shaken, the clamp at *c* is then closed, *M* and the glass tube *s* removed, and *r'* is brought below the surface of a known quantity of water contained in a tall beaker. The clamp is again opened, and as the liquid runs back into *G*, a measured amount of water is poured into the beaker till the level is the same in both vessels, when *G* and the generator are evidently once more under ordinary atmospheric pressure.

The volume of water which has been drawn back into *G* out of the beaker, deducted from that which has passed from *G* into *M* during the decomposition of the carbide, thus gives the volume of acetylene generated; and this is corrected for temperature, pressure, and vapour tension of water in the usual way.

Two samples of Neuhausen carbide gave as the mean of five tests each 286·8 and 297·6 litres per kilogramme (4·59 and 4·88 cubic feet per lb.). F. H. L.

The Analysis of Commercial Calcium Carbide and Acetylene. G. Lunge and E. Cedercreutz. (*Zeit. angew. Chem.*, 1897, 651-655.)—The gas produced on adding water to commercial calcium carbide is never pure acetylene, but contains impurities up to 4 per cent., including sulphuretted hydrogen, phosphuretted hydrogen, ammonia, carbonic oxide, hydrogen, nitrogen and oxygen. Of these the most important are the two first. In good calcium carbide the amount of calcium phosphide and calcium sulphide is very trifling; but the authors have met with a sample containing so much of the former that the gas liberated ignited spontaneously.

In the analysis a sample of from 50 to 100 grammes, as representative of the bulk as possible, is decomposed with water, and the gas conducted into a laboratory gasometer holding not less than 40 litres, and the water in which is saturated with acetylene. Here it is measured and reduced to standard temperature and pressure. Theoretically, 100 grammes of pure calcium carbide yield 40·625 grammes of acetylene (= 34·877 litres), and commercial carbide ought to produce not less than 300 litres of gas per kilo.

For the determination of the phosphuretted hydrogen the ordinary method of oxidation with bromine water is not applicable, on account of the strong action of bromine on acetylene, and the authors therefore employ a solution of sodium hypochlorite as the oxidizing agent. From 50 to 70 grammes of the carbide in lumps are decomposed by water, which is allowed to fall drop by drop from a stoppered funnel passing through the cork of the flask. The outlet tube of the flask is connected with a tube composed of a series of bulbs, in which are placed 75 c.c. of a 2 to 3 per cent. solution of sodium hypochlorite. After the whole of the carbide has been decomposed, which takes from three to four hours, the flask is filled to the neck with water, a little air drawn through the apparatus, the contents of the bulb-tube emptied into a beaker, and the phosphoric acid determined by precipitation with magnesia mixture.

The sulphur, which has been simultaneously oxidized, can be determined in the filtrate from the ammonium magnesium phosphate precipitate by acidifying with hydrochloric acid and precipitating with barium chloride. C. A. M.

The Commercial Analysis of Sulphur Chloride. G. A. Le Roy. (*Rev. de Chim. Indust.*, 1897, viii., 294, 295.)—The chief industrial uses of sulphur chloride are in the vulcanizing of rubber, the preparation of artificial indiarubber, and the treatment of minerals containing gold. It contains normally 47 per cent. of sulphur and 53 per cent. of chlorine, but in many industrial samples the proportions are very different from this. Thus, in several the author found as much as 10 per cent. of uncombined sulphur in solution, whilst in others there was a large excess of free chlorine.

In Rose's method of analysis the sulphur is estimated by oxidation with fuming nitric acid and precipitation as sulphate, and the chlorine determined by difference. In place of this, the author describes the following volumetric method: From 10 to 12 grammes of the sample (roughly measured) are placed in a graduated flask

containing a known weight of crystallizable benzene; the flask and its contents are weighed after the addition, and the difference gives the amount of sulphur chloride taken. The liquid is made up to the mark with benzene, and an aliquot portion used in the determination.

The fraction taken is shaken with a known volume of standard soda, containing 19·37 grammes Na_2O per litre (1 c.c. = 0·01 gramme sulphur), which decomposes it into the sulphide, sulphite, hyposulphite, sulphate and chloride of sodium, with the liberation of some sulphur in the free state. The flask is then warmed on a sand-bath, which causes the benzene to evaporate and the sulphur to dissolve in the soda. When the sulphur has completely dissolved, an excess of hydrogen peroxide is added. The latter must be neutral and free from substances which can be precipitated by silver nitrate. On warming the liquid, the various compounds of sodium and sulphur are converted into sulphate. The excess of hydrogen peroxide is removed by boiling, or, if necessary, by the addition of several drops of permanganate solution, and after cooling the excess of soda is titrated with normal nitric or sulphuric acid. The quantity of soda left free, plus the soda in combination with the chlorine subsequently determined, deducted from the total amount of soda taken, gives the number of c.c. equivalent to the soda converted into sulphate. From this the amount of sulphur is obtained by multiplying by 0·01.

In the liquid thus neutralized, the chlorine is determined by titration with a silver solution by Mohr or Volhard's method. C. A. M.

Use of Caustic Alkalies in a Powdered Condition. A. Seyda. (*Zeit. für öffent. Chemie*, iii., 438.)—Powdered caustic alkali is recommended for use in the preparation of alcoholic saponifying solutions, etc. The quantity required is weighed approximately in the form of stick caustic, and is then finely powdered and dried in an exsiccator over sulphuric acid. Alkali in this powdered condition dissolves almost immediately in alcohol. The author uses powdered sodium hydrate prepared in this way in his method of testing organic preparations for iodine (ANALYST, vol. xxii., p. 325), and suggests its use for many other purposes where fusion with soda is required. H. H. B. S.

APPARATUS

A Combustion Boat with Partitions. E. Murmann. (*Zeit. anal. Chem.*, 1897, xxxvi., 380-381.)—In the ordinary boat the melted substance collects in the cooler end where the entire mass is suddenly decomposed when the decomposition temperature is reached. This is obviated by the use of a partitioned boat, in which only a small portion of the substance is decomposed at a time. The boats are made of porcelain, with ten divisions, and may be obtained in two sizes (70 mm. by 10 mm. and 100 mm. by 13 mm.) from Lenoir and Forster, Chem. Phys. Institut. Vienna.

C. A. M.

THE ANALYST.

FEBRUARY, 1898.

PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS.

THE ANNUAL DINNER OF THE SOCIETY OF PUBLIC ANALYSTS.

THIS, the full report of which will appear in our next issue, took place at the Criterion on Tuesday, the 18th ult., the President (Dr. Bernard Dyer) occupying the chair. The Annual Meeting was held on February 2nd.

REPORT OF THE COUNCIL OF THE SOCIETY OF PUBLIC ANALYSTS ON THE SALE OF FOOD AND DRUGS BILL,

INTRODUCED IN THE HOUSE OF COMMONS IN 1897

BY THE

RIGHT HON. W. H. CHAPLIN, M.P., AND MR. T. W. RUSSELL, M.P.

THE Council of the Society of Public Analysts has considered the Sale of Food and Drugs Bill, introduced by the President and Parliamentary Secretary of the Local Government Board towards the close of the last Session, and desires to express its regret that the measure deals with so few of the recommendations of the Select Committee. It is to be remembered that the Select Committee of the House of Commons on Food Products Adulteration, which was appointed in 1894, sat for three Sessions, and collected a very large mass of evidence, from widely different sources, bearing upon the matters which it was appointed to consider. The outcome of its labours was a report, presented to Parliament in July, 1896, very fully summarising the evidence which had been collected, and concluding with an epitome, under twenty-three headings, of the directions in which, in the opinion of the Select Committee, the existing Food and Drugs Acts should be amended. It was confidently expected on all hands that the Government would introduce into Parliament a Bill giving effect to these recommendations, or at all events to the majority of them ; but the Bill produced at the close of the last Session falls so far short of the requirements of the case, that in most essential particulars the recommendations of the Committee appear to have been passed over.

The Council of the Society of Public Analysts desires, therefore, to point out the

chief omissions in the Bill, and the principal directions in which it appears to require amendment.

The First Section of the Bill states that—

> "It shall be the duty of every local authority entrusted with the execution of the laws relating to the sale of food and drugs, to put in force from time to time, as occasion may arise, the powers with which they are invested, so as to provide proper securities for the sale of food and drugs in a pure and genuine condition, and in particular to direct their officers to take samples for analysis."

It appears to the Council that this Section gives no remedy for the existing unsatisfactory state of affairs, no means being provided or suggested for enforcing the performance of that which is recognised as a duty. Experience has shown, as is fully set forth in the Report of the Select Committee, that in spite of considerable pressure brought to bear by the Local Government Board, certain local authorities have persistently neglected to carry out the provisions of the Sale of Food and Drugs Acts.

The Second Section, which proposes to institute a *minimum* fine of £5 for a second or subsequent offence, carries into effect recommendation No. 16 in the Report of the Select Committee. This is one of the very few of the recommendations of the Committee which the Council feels itself unable to support, since the Council of the Society of Public Analysts is of opinion that the fixing of a minimum fine of £5, in all cases of second conviction, would be unduly binding on magistrates, who should be free to deal with each case on its merits, having regard to the great variety of circumstances requiring the consideration of the Bench which occur in connection with cases under the Acts.

With regard to the Fourth Section, the Council desires to draw attention to a recent case in which margarine was enclosed in a wrapper on which the word "margarine" was distinctly and legibly printed, but in which the significance of the word was diminished by the presence of other printed matter on the label. The Council therefore suggests that this Section might be improved by adding a provision that no other words or letters should be printed on the same wrapper.

With regard to Section 10, which relates to warranties, the Council of the Society of Public Analysts considers that, in order to ensure the conviction of the real offender in a case of adulteration, jurisdiction should be given to the Court before which the summons is heard, to call the giver of a warranty before it, should the warranty exonerate the vendor; and that proceedings should in such case be compulsorily instituted against the giver of the warranty. The Council also considers that no warranty should be accepted if given by a person abroad, or outside of such jurisdiction as it is possible to confer on the Court by Act of Parliament.

Section 12 (Sub-Section 1) proposes that the certificate of analysis required by the Sale of Food and Drugs Act of 1875 shall be in a form prescribed by the Commissioners of Inland Revenue, and proposes to repeal the Schedule to that Act relating to the form of certificates.

The Council of the Society of Public Analysts is of opinion that the form of certificate should not, as in the Act of 1875, be unalterably fixed, but dissents from

the proposal that the form should be prescribed by the Commissioners of Inland Revenue. The Council considers that the proper body to prescribe the form of certificates would be the Standing Committee or Court of Reference recommended by the Select Committee, the formation of which Court of Reference is not provided for in Mr. Chaplin's Bill, an omission to which attention is called hereafter.

Sub-Section 2 of Section 12 provides that the production by the defendant of a certificate by a public analyst shall be sufficient evidence of the facts therein stated (unless the prosecutor requires that such public analyst be called as a witness). The Council would suggest the desirability of providing that in the absence of the public analyst whose certificate is tendered by the defendant, and in the event of a material difference between the opinions expressed in that certificate and in the public analyst's certificate on which the prosecution was based, it should be compulsory on the Court to refer the portion of the sample retained by the Inspector to the Commissioners of Inland Revenue.

Having thus referred to some of the points suggested by the Government Bill, the Council wishes to call attention to certain of its omissions. The Select Committee made an important recommendation (No. 18)—

> " . . . . that the definition of the word 'food' as used in the Acts should be amended so as to include expressly all articles intended to enter into or be used in the preparation or flavouring of food."

This recommendation has not been adopted in the Government Bill, although its importance was very fully insisted upon. The present definition of food, as interpreted by the High Court, enables injurious, adulterated, or objectionable baking powders and certain other articles, to be sold with impunity, on the contention that they are not themselves "food," although they are sold for the express and avowed purpose of being introduced into food and consumed as such.

The most important omission, however, in the opinion of the Council of the Society of Public Analysts, is the failure to carry out recommendation No. 19 of the Select Committee, viz. :

> " . . . . that an authority should be constituted, who should act as a Court of Reference upon scientific and other questions arising under the Acts, and who should be empowered at their discretion to prescribe standards and limits of the quality and purity of food."

The report of the Select Committee deals with a very large number of questions on which, as a Committee, they refrain from making any definite recommendations, on the ground that such matters would properly be dealt with by the body whose formation they contemplated. They expressed the opinion—

> " . . . . that it would greatly facilitate the working of the laws as to the adulteration of food, and also serve the interests of traders, if standards or definitions of food were promulgated by competent authority"; and
> " . . . . that the question of fixing food standards should receive the attention of a specially-constituted scientific body familiar with questions of analysis and the chemistry of food."

Their recommendation was that such body or Court of Reference should—

> " . . . . be empowered to make such orders as they think proper respect-

ing standards of the quality and purity of food, and that such orders should, when confirmed by a Secretary of State, have the force of law"; and they were of opinion that ". . . . the formation of such an authority having the functions indicated would result in the removal of many practical difficulties met with in the administration of the Acts, and by rendering more precise the law affecting adulteration would obviate the costly litigation in which, under existing circumstances, traders and others have sometimes been involved."

It was recommended that the proposed—

" . . . . Court of Reference should take the form of a standing Departmental Committee appointed by the Board of Trade;" and that " . . . . such Committee should include the principal officer of the Government laboratory, nominees of the Local Government Board and Board of Agriculture, one or more analysts of repute, and representatives of the General Medical Council, the Institute of Chemistry, and the Pharmaceutical Society. Other scientists," it is added, "whose services are available, and whose presence on the Committee may be expected to lend weight to its decisions, should find places thereon."

It is further suggested that:

" . . . . representatives of the trading and manufacturing community should also be included."

In fact, this proposal as to the establishment of a Court of Reference having such powers as those alluded to, forms, from most points of view, one of the principal features in the report of the Select Committee; and the recommendation met with cordial and widespread endorsement. This being so, the Council of the Society of Public Analysts notes with very great regret the total omission from the Bill of any mention of such a Court or Committee. It is, in the opinion of the Council, essential for the success of any new legislation, not only that such a Court or Committee should be constituted, but that the Committee when formed shall include one or more *public* analysts. At the present time each individual public analyst is left to decide what does or does not constitute adulteration, what is or is not to be regarded as injurious to health, and what limits and standards are to be adopted in judging of the genuineness or otherwise of samples submitted to him. Disputed questions of the greatest scientific intricacy are now fought out, more or less imperfectly, in open Court at the expense of vendors or public authorities; and, as a consequence, questions which might have been settled, to the benefit of the community, many years ago, are still undecided, after more than twenty years' working of the Food and Drugs Acts.

The Court of Reference recommended by the Select Committee was originally suggested by the Society of Public Analysts, and the suggestion, as will have been seen from the quotations already given from their report, met with the entire approval of the Select Committee, who saw therein the means of solving the gravest difficulties which have hitherto arisen under the Food and Drugs Acts. The Council is clearly of opinion that, without such a Court or Standing Committee, any new legislation will be of little or no value, while, on the other hand, the institution of such a

Committee would afford the simplest means of ensuring the effective working of anti-adulteration legislation.

There are other points of omission in the Bill (such, for instance, as those connected with the Select Committee's recommendations affecting the colouring and mixing of butter substitutes), which the Council of the Society of Public Analysts does not feel itself directly called upon to discuss. Generally speaking, the Council is of opinion :

1. That the draft Bill introduced by the President and Secretary of the Local Government Board is unsatisfactory, inasmuch as it does not embody the most important recommendations of the Food Products Adulteration Committee, and that, if passed, it would not lead to the removal of the difficulties experienced in carrying out the present Sale of Food and Drugs Act.
2. That the recommendations of the Food Products Adulteration Committee, subject to but few modifications, should be embodied in any Bill which may be put before Parliament during the coming Session.
3. That the most important recommendation of the Select Committee, viz., recommendation No. 19, referring to the formation of a Court of Reference constituted substantially as laid down in the body of the report (with, however, the inclusion in its number of one or more public analysts) should be acted upon, if new legislation is to lead to satisfactory results.

(Signed) BERNARD DYER, *President.*
(Signed) EDWARD BEVAN, } *Hon. Secretaries.*
(Signed) CHARLES E. CASSAL, }

A METHOD OF ESTIMATING GALLOTANNIC ACID BY MEANS OF THE POLARIMETER.

By R. F. Wood-Smith and Cecil Revis.

(*Read at the Meeting, December* 1, 1897.)

It is a well-known fact that difficulty has often been experienced in the Löwenthal method of estimating tannic acid, in properly filtering the gelatino-tannin precipitate, the filtrate from which is apt to remain turbid and to contain traces of tannic acid ; the method offers the further objection that it is not always easy to obtain a sensitive final reaction in the permanganate titration.

The method proposed by the authors removes these objections, and has the advantage of great rapidity ; but, at the same time, it must be borne in mind that it is based upon empirical data, that the experiments made were carried out in the case of gallotannic acid only, and that therefore varying results must be expected where other tannin material is employed.

The method consists in observing the lævo-rotation of a standard gelatin solution before and after treatment with known quantities of tannin material (the filtered liquids in each case being clarified by egg-albumin), when, at a constant

temperature, the quantity of gallotannic acid present (in the absence of other bodies optically active towards the polarized light) can be determined by a factor for the rotation difference.

It was found that at constant temperature the specific rotatory power of gelatin remained the same for any strength of solution up to 10 per cent., and it was further found that the difference between the rotatory power of the gelatin solution before and after treatment with tannin at constant temperature was directly proportional to the amount of gallotannic acid used, so long—and this was observed to be a most important condition—as sufficient excess of gelatin remained after treatment to cause a lævo-rotation of not less than seven divisions on the Ventzke scale.

The experiments for the determination of the factor were all carried out with a Schmidt and Haensch half-shadow quart-compensating polarimeter, an instrument which is constructed for use with "white light," as it was believed by the authors that this form of instrument was most generally employed for rapid work.

The gelatin used for the experiments, and for which the factors are calculated, is the well-known Coignet's gold label extra fine gelatin, since this was found to be constant in composition and in its specific precipitating power on gallotannic acid. It is extremely important that gelatin of this nature should be employed, since, as has been recently pointed out by P. Carles (*Ann. de Chim. Analyt.*, vol. ii. [10], 181, 182), different kinds of commercial gelatin precipitate very different quantities of gallotannic acid under the same conditions, and also possess different specific rotatory powers.

The temperature at which the experiments were carried out was 45° C., since at that point the specific rotatory power of gelatin only varies very slightly with changes of temperature, and it was found that differences of 3° or 4° produced no appreciable difference in the final results.

The gelatin solutions were also made up and kept at that temperature in a water-jacketed incubator, and all volumetric measurements occurring in the course of the experiments were made under the same conditions.

The details of the method and the calculated factors are here given:

A 1. 25 c.c. of an approximately 9 per cent. solution of gold label gelatin in water at 45° C. are transferred to an evaporating dish capable of holding about 100 c.c., which is placed on a boiling-water bath.

A 2. As soon as the temperature of the liquid has risen to about 60° C., 1 c.c. of the soluble albumin of a perfectly fresh egg is added, and the whole is allowed to attain the temperature of the water-bath, with constant stirring.

A 3. The liquid is kept at 100° C. for ten minutes, also with repeated stirring.

A 4. It is now allowed to cool down to about 50° C., when, if the supernatant liquid is seen to be quite clear, it is again heated up to 100° C. and filtered, the precipitate washed with boiling water, and made up to 50 c.c. at 45° C.; but if, as sometimes happens, the first treatment with albumin does not render the fluid free from opalescence, the above operation is repeated, when a clear liquid can always be obtained, especially if the warming on the water-bath be slightly prolonged.

A 5. It is advisable, if the bulk of liquor has during the process attained unduly large proportions, not to evaporate down, but to filter, wash as far as possible, and

make up to 50 c.c., and then to again wash the residue with boiling water, and make up the second washing to another 50 c.c.

A 6. The lævo-rotation of the clear filtrate is now taken at 45° C., to which must be added that of the second washing if necessary.

B 1. 25 c.c. of the same 9 per cent. gelatin solution are, as before, transferred at 45° C. to an evaporating dish, and a known quantity of the tannin extract added (which should for convenience contain between ·2 and 1 gramme of gallotannic acid).

B 2. The above is warmed on the water-bath for five to ten minutes, when the opalescent supernatant liquid is poured off into a second dish, the gelatino-tannin precipitate which adheres to the first basin being thoroughly worked about and washed with boiling water, and the washing added to the second basin.

B 3. This is now treated with egg-albumin in the same manner as the blank experiment (A 2-6), and is then filtered, made up to 50 c.c., and the lævo-rotation at 45° C. taken.

The difference between the rotations of A and B gives the measure of the gallotannic acid present. The experiments A standardize the gelatin solution, and the rotation so found may be taken as constant for the same solution for some time.

The following table states the gelatin differences of rotation caused by different amounts of gallotannic acid, and the factor which must be multiplied by the sugar units of difference (for white light) to give the amount of gallotannic acid present:

Weight of Gallotannic Acid Used = T.	Gelatin Rotation Difference in Sugar Units = R.	Factor $= \frac{T}{R}$.
0·900 grammes	13·50	0·0667
0·720 ,,	10·60	0·0679
0·600 ,,	8·90	0·0674
0·480 ,,	7·15	0·0671
1·000 ,,	14·90	0·0671

Mean factor $\frac{T}{R}$ for the above and numerous other experiments = 0·0673.

Addenda.—(1) It is important that the egg employed for clearing the liquid should in all cases be *perfectly* fresh, as otherwise the albumin does not coagulate properly, and the filtrate retains a slight opalescence.

(2) To ensure rapid filtration, a fluted filter should be employed; then the process takes place quickly, and a hot-water funnel is not necessary.

(3) After the tannin material has been added to the gelatin solution and warmed, the supernatant liquid should appear quite milky, this being an evidence that a sufficent excess of gelatin exists.

Discussion.

Mr. Hehner inquired whether the method was applicable to more than one kind of tannin, and, if not, to what particular kind of tannin it was applicable. It appeared to have the same difficulties as all other methods—in being useful only for one of the very numerous kinds of tannic acids. What was to be desired was a method for actually determining tannin, from whatever source it came.

Mr. ALLEN observed that a single method for the determination of tannin in any class of tanning material was as far out of reach as a universal method for the determination of all metals, for the variations in the properties of the so-called "tannins" were quite as great as the differences in the analytical behaviour of, say, iron, copper, tin, and zinc.

Dr. RIDEAL agreed with Mr. Hehner and Mr. Allen as to the variations in different tannins, and as to the difficulty of determining them. He had himself been in the habit of determining the quantity of potassium permanganate required by a solution of tannin (Loewenthal's method), and the quantity required by the tannin solution after treatment with hide-powder, representing the quantity of organic matter in the solution before and after treatment, the difference between the two being the actual weight of organic matter present as tannin, or matter absorbable by hide-powder. The use of a very pure gelatin seemed to have an advantage over hide-powder, inasmuch as there was a good deal of difficulty in obtaining uniform hide-powder. The use had been recently suggested of a compound of gelatin and formalin, which absorbed tannin in the same way as gelatin, and which might possibly have a more constant composition.

Mr. A. C. CHAPMAN thought Mr. Allen's remark too sweeping. It was perfectly well-known that there were different kinds of tannin, which behaved differently in regard to their action on gelatin; but if separate processes could be devised to give accurate or approximately accurate results with, for example, bark, tea, hops, and like materials respectively, each of these processes would be extremely useful, even if it were not applicable to more than one kind of material.

The PRESIDENT observed that the method, although it might not be applicable to different classes of tannin equally, was certainly an interesting one, and one which opened up new avenues of investigation.

Mr. WOOD-SMITH admitted that the method was only applicable for the particular kind of tannin employed, viz., the ordinary purified commercial gallo-tannic acid, and that, further, the presence of optically-active bodies other than tannic acid necessarily interfered with the results. It had, however, proved very useful to the authors under circumstances involving the use of one class of tannic acid only, and factors could no doubt be determined for any other variety of tanning material. In reply to a question put by Mr. Richmond, he said that the normal weight of sugar solution for the polarimeter employed in the experiments was 26·048 grammes.

A BUTTER EIGHTEEN YEARS OLD.

By E. G. CLAYTON.

(*Read at the Meeting, December* 1, 1897.)

THE butter to which these remarks apply was the subject of a reference-case under the Sale of Food and Drugs Act in 1879. In that year it was analysed by Mr. Hehner, who has kindly given me permission to mention that he found it to yield 87·75 per cent. of insoluble fatty acids, while it contained normal amounts of water, curd, and salt, and evidently was perfectly genuine.

Since 1881 the bottle containing the residual portion of the sample has been in my keeping, and the following are the results of the examination to which I have submitted this old butter. It should be premised that the quantity was very small, otherwise a more complete investigation would have been made. The sample had rather an offensive odour, and was considerably discolored. During most of the time the bottle was kept in a dark cupboard, but the stopper, being broken, fitted imperfectly, so that there was ample opportunity for oxidation.

ANALYSIS:

Melting Point.	Density at 100° C. (Water at 15·5° C.).	Insoluble Fatty Acids.	Soluble Fatty Acids.	Reichert number (5 grammes used).	Koettstorfer numbers.	Hübl (Iodine absorbed).	Maumené.	Rancidity.
33° C.	·8742*	85·72%*	7·36%	22·36 c.c.* $\frac{N}{10}BaH_2O_2$.	Required 23·47% KOH; = 239·0, saponification equivalent.	(*a*) Estimated in 1895: 25·68%.* (*b*) Estimated in 1897: 25·09%.	Rise of temperature = 22°C.	100 grms. required 160·3 c.c. normal KOH.

The numbers with asterisks were obtained by me in January, 1895, the remainder in October, 1897.

ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

FOODS AND DRUGS ANALYSIS.

The Adulteration of Milk with Saccharose Solutions. Cotton. (*Bull. de Pharm. de Lyon*, August, 1897; through *Jour. Pharm. Chim.*, 1897, vi., 362, 363.)—The following is given as a rapid test for detecting the addition of saccharose solutions to milk: Ten c.c. of the suspected milk are mixed with 0·5 gramme of powdered ammonium molybdate, and 10 c.c. of dilute hydrochloric acid (1 : 10). In a second tube 10 c.c. of milk of known purity, or 10 c.c. of a 6 per cent. solution of lactose, are similarly treated. The two tubes are placed in a water-bath, and the temperature gradually raised. At about 80° C. the milk, if adulterated with saccharose, assumes an intense blue colour, whilst the genuine milk, or lactose solution, remains unaltered. On boiling, these also turn blue, but to less extent than the adulterated milk.

With milk containing only 1 gramme of saccharose per litre, the reaction is well marked, but the author states that the amount used for adulteration is never less than 6 grammes per litre. C. A. M.

The Composition of Asses' Milk. A. Schlossmann. (*Zeit. Phys. Chem.*, 1897, xxiii., 258).—The author gives the results of his analyses of sixteen samples obtained from one establishment in Dresden. The density varied from 1·031 to 1·036, and the mean percentage results of the total solids were 11·15; of the ash, 0·399; and of the sugar, 4·94. The total nitrogen varied from 0·22 to 0·27 per cent., of which 86 per cent. belonged to albuminoid substances (precipitated by trichloracetic acid or salicyl-sulphonic acid). The limits of fat, of which the globules were very small, were 0·15 and 0·6 per cent. One c.c. of the milk required for neutralization 0·06 c.c. of decinormal sulphuric acid with phenol-phthalein as indicator, and 0·404 c.c. with methyl orange or turmeric as indicator. C. A. M.

On the Volumetric Determination of Casein in Milk. G. Deniges. (*Jour. Pharm. Chim.*, 1898, vii., 9-12.)—This is a supplementary note on the author's volumetric process (ANALYST, xxii., 11). In the titration of milk rich in calcium salts, a turbidity occurs after the addition of ammonia which prevents the end-reaction from being clearly observed. This is obviated by the addition of a little ammonium oxalate, which does not exercise any disturbing influence. The modified process is as follows: Twenty-five c.c. of the milk are mixed with 5 c.c. of a saturated solution of ammonium oxalate, 20 c.c. of the decinormal mercuric-potassium iodide solution, and 2 c.c. of acetic acid, the volume made up to 200 c.c. and the liquid filtered. To 100 c.c. of the clear filtrate are added 10 c.c. of a solution of potassium cyanide equivalent to a decinormal (or approximately decinormal) solution of silver nitrate, and 12 to 15 c.c. of ammonia, and the liquid titrated with the silver nitrate solution until there is a permanent turbidity. The number of c.c. used = q.

A blank determination is made to determine the exact value of the silver nitrate solution. Ten c.c. of the cyanide solution, 12 to 15 c.c. of ammonia, and 10 c.c. of the mercuric-potassium iodide solution, are mixed with 100 c.c. of water and titrated as before with the silver nitrate. The number of c.c. = c, and the casein in the milk corresponds to $q - c$. When the silver nitrate is exactly decinormal, $c = 4·8$ c.c. The value of $q - c$, expressed in tenths of a c.c., is then obtained from the table or the formula given in the former paper. C. A. M.

A Contribution to the Study of Butter. K. Farnsteiner and W. Karsch. (*Zeit. für Untersuch. der Nahrungs-und Genussmittel*, 1898, 16-21.)—The authors call attention to a case of butter of unquestionable purity showing abnormally low Reichert-Meissl and saponification numbers. Different samples of the butter, which came from a farm in Holstein, gave the following numbers:

Refractometer number (25° C).	Reichert-Meissl number.	Saponification number.
55·0	23·98	220·27
55·0	23·68	219·20
55·0	22·80	218·53
55·0	22·74	218·50

The case was further inquired into, and 25 cows out of the 180 kept at the farm were selected as corresponding to the whole number as regards the average period of

lactation. The milk from these being treated separately, yielded a butter which gave the same abnormal results, viz. :

Refractometer number. (25° C).	Reichert-Meissl number.	Saponification number.
53·0	22·16	220·40
53·2	22·22	221·75
53·0	22·11	219·74

The relation of the refractometer number to the iodine number is shown in the following figures :

	Refractometer number.	Iodine number.	Reichert-Meissl number.	Saponification number.
October 21, from 180 cows	55·0	49·57	22·80	218·53
October 26, ,, 25 ,,	53·0	40·00	22·11	219·74

The case corresponds to that reported from Hameln by Vieth, and is doubtless due to the cows having been for a long time in milk. H. H. B. S.

Detection of Sesame-oil in Butter and Margarine. P. Soltsien. (*Pharm. Zeit.*, 1897, xlii., 837 ; through *Chem. Zeit. Rep.*, 1897, 325.)—The author calls attention to the fact that when furfuraldehyde, or at least the German official alcoholic solution thereof, is mixed with hydrochloric acid (sp. gr. 1·19), it yields of itself a red coloration, which appears slowly in the cold, but quickly on warming ; and the tint is very similar to that produced by a small amount of sesame-oil. The only difference between the two is that the former develops more slowly, soon changes to the colour of permanganate, and finally assumes a bluer tone, which is fairly permanent. F. H. L.

On the Detection of Ergot in Flour. A. Miller. (*Jour. Soc. Phys. Chim.*, xxviii., 824 ; through *Jour. Pharm. Chim.*, 1897, 553.)—The author has met with a sample of flour containing only a trace of ergot (not more than 0·1 per cent.), which, nevertheless, imparted to the alcoholic extract a clear rose coloration as pronounced as though the proportion of ergot had been 1 per cent. On examination, the flour was found to contain particles of the husk of the grain of a bluish-green shade, which assumed a reddish tint on treatment with acidulated alcohol, and imparted the same colour to the alcoholic extract. The difference in shade between the colour produced by this flour and that containing ergot was only distinguishable in concentrated solutions, when the former was rose-red whilst the latter was brick-red. C. A. M.

TOXICOLOGICAL ANALYSIS.

On the Detection of Phosphorus in Toxicological Work. H. Natterman and A. Hilger. (*Forsch. Ber.*, 1897, iv., 241-258.)—The methods employed for the detection of phosphorus in forensic work are : (1) Scherer's, which depends upon the fact that steam containing phosphorus vapour reduces silver nitrate. The substance under examination is mixed with water, acidified with sulphuric acid, and warmed at 30° to 40° C., whilst strips of the reagent paper are suspended in the mouth of the

flask. (2) Mitscherlich's, which is based on the volatility of elementary phosphorus in steam, and its becoming luminous on contact with oxygen. (3) Dussart-Blondlot's, which is especially suitable for the detection of phosphorus in an oxidized state. It depends on the fact that phosphorus, and many of its compounds, when treated with nascent hydrogen, yield a gas which, when ignited, gives a flame the inner cone of which is of a deep-green colour.

With regard to Scherer's reaction, the author points out that many substances may cause a darkening of the silver nitrate paper, and that the test must be regarded as a negative one (the absence of any discoloration proving the absence of free phosphorus) rather than as a positive one, unless phosphoric acid can be identified in the blackened paper.

In Mitscherlich's test the experiments described at length in the paper show that in the distillation of the phosphorus in the ordinary way the amount recovered is only a fraction of the whole, though the yield increases as the quantity of phosphorus present becomes larger. This is largely due to a certain amount of oxidation taking place within the flask and the non-volatility of the oxidation products. To obviate this the author has devised a modification of the process, in which a current of carbon dioxide is led into the flask during the distillation. The steam carrying the phosphorus passes upward through a narrow tube which is bent twice at right angles, first horizontally, then downwards, is condensed in a vertical condenser, and falls into an Erlenmeyer flask. Connected with the side-tube of the flask is a suction-pump, by means of which air is drawn into the horizontal part of the narrow tube through a smaller tube fused on, and closed when not in use by an indiarubber tube and spring clamp.

In this way the oxidation takes place entirely in the tube, whilst the interior of the distillation-flask remains absolutely dark, and it is possible to recover in the distillate over 90 per cent. of the phosphorus present in the original substance. The smallest quantity of phosphorus capable of detection by the Mitscherlich process is 0·00006 gramme.

Where oxidation is prevented by drying, or by the course of putrefaction, a small amount of free phosphorus can be detected in organic matter long after it has been mixed with it. Thus, 0·003 gramme of phosphorus was mixed with organic matter in a state of putrefaction, and could still be detected after a lapse of six months.

In the Dussard-Blondlot test certain substances, noticeably ether and proteid derivatives, interfere with the reaction. This is obviated by passing the phosphuretted hydrogen first through potash to remove sulphuretted hydrogen, and then into silver nitrate solution and applying the test to the precipitate of phosphor silver.

The author recommends the following modification of the test as more simple and sensitive than the ordinary process: The filter containing the silver phosphide precipitate is placed, together with a little pure zinc and some water, in a small flask fitted with a cork with three perforations. Through two of these pass tubes bent at right angles, whilst a third is for a thistle funnel. One of the tubes is connected with a Kipps' hydrogen apparatus, the gas generated in which is proved by the spectroscope to be free from phosphorus, whilst the other tube is connected with a U-tube filled with pumice saturated with potash, and in which is inserted the platinum jet.

Hydrogen is then generated within the flask by introducing through the thistle funnel a few c.c. of dilute sulphuric acid (1 : 5), and this is swept onwards by means of the current of hydrogen from the Kipps' apparatus, and a strong flame is obtained at the jet. The smallest amount of phosphorus capable of detection by this reaction with the naked eye is 0·00000006 gramme, but even less than this can be recognised with the spectroscope.

The chief drawback to the process is the length of time required to reduce phosphorus in the form of acids to phosphuretted hydrogen. From ten to fourteen days are required for the reduction of only a few milligrammes, and then never more than one-fifth of the total amount present is converted. The author finds, too, that the precipitate of silver phosphide does not account for the whole of the phosphorus, and that the solution of silver nitrate often contains as much as the washed precipitate. He suggests that advantage may be taken of the complete absorption of phosphuretted hydrogen in a solution of copper chloride. Thus, a solution prepared by dissolving 4 grammes of pure copper chloride in 20 c.c. of hydrochloric acid (sp. gr. 1·19), adding 30 c.c. of a 12 per cent. solution of potassium hydroxide, and pouring off the liquid from the crystals separating, absorbs the gas completely, and readily parts with it again on warming. C. A. M.

ORGANIC ANALYSIS.

Estimation of Small Quantities of Methyl Alcohol, Formaldehyde, and Formic Acid. Nicloux. (*Bull. Soc. Chim.*, 1897, xvii., 839, 840.)—This is an extension of the author's process for determining ethyl alcohol in very dilute solution, based on the reduction of potassium bichromate by the alcohol in the presence of sulphuric acid (*Bull. de la Soc. Therap.*, iii., 860). In each case the solution under examination must contain only one of these bodies, and must also be free from other organic substances capable of reducing bichromate. In the determination, 5 c.c. of pure concentrated sulphuric acid are added to 5 c.c. of the solution, the mixture heated for a minute and titrated with standard bichromate (the strength of which varies according to the substance to be determined) until a green tint is obtained. It is advisable to compare the colour with that of a series of standard colour samples recently prepared so as to obtain the exact value of the standard solution.

The strength of the bichromate solution is : for methyl alcohol, 19 grammes ; for formaldehyde, 17 grammes ; and for formic acid, 11 grammes of bichromate per litre respectively. In each case 2 c.c. corresponds to 5 c.c. of a solution containing 1 gramme of the substance per litre (*cf.* ANALYST, xii., 25 and xxii., 161 and 263).

C. A. M.

On the Detection of Formalin. A. Jorissen. (*Jour. Pharm. de Liège.* 1897, iv., 257 ; through *Bull. de la Soc. belge*, 1897, 211, 212.)—The author gives a summary of different tests used for the detection of formalin, but objects to all of them with the exception of Hehner's reaction (ANALYST, xxi., 95) on the ground that they are not peculiar to formaldehyde. He finds that in the case of the last it is possible

to replace the milk by certain alkaloids, of which several give characteristic reactions of extreme sensitiveness. Thus, on placing a little morphine hydrochloride in a porcelain basin, adding a few drops of concentrated sulphuric acid, and touching the mixture with the end of a glass rod previously dipped in a solution of formaldehyde, even if exceedingly dilute, a purple colour is produced which changes to an indigo blue. C. A. M.

Analysis of Fats. Extraction of Cholesterin and Phytosterin. A. Bömer. (*Zeit. für Untersuch. der Nahrungs-und Genussmittel*, i., 21-49.)—The following process is recommended: 50 grammes of fat are saponified with 100 c.c. of alcoholic potash solution, the solution diluted with 200 c.c. of water, and the whole shaken for from a half-minute to a minute with 500 c.c. of ether. After standing for two or three minutes, the etheric extract is separated and distilled. The soap solution is then again extracted two or three times with 200 to 250 c.c. of ether, each extract being distilled off as before. After the ether has all evaporated, a little alcohol usually remains in the residue, and this is removed by blowing air through it, while the vessel containing it is immersed in a bath of boiling water. The residue still contains a little unsaponified fat, which is removed by a further saponification with 10 c.c. of alcoholic potash. The contents of the flask are now washed out into a separating funnel with 20 c.c. of water, 80 to 100 c.c. of ether added, the whole shaken for half a minute, allowed to stand for a few minutes to separate, the aqueous-alcoholic portion drawn off, and the etheric extract washed three times with 5 to 10 c.c. of water. After removal of the last wash-water, the etheric extract is filtered into a small beaker or Erlenmeyer flask, and the ether slowly evaporated off. On drying in the water-bath, a crystalline residue is obtained containing the cholesterin and phytosterin, from which these bodies can be obtained in a pure state by crystallization.

Cholesterin can be easily distinguished from phytosterin by the form of the crystals and the manner of its crystallization. If, however, both bodies are present together, the mixture crystallizes in one form only, the crystals either approximating to the form of phytosterin crystals, or, if cholesterin is present in the greater quantity, differing in form from the pure crystals of either body. H. H. B. S.

On the Cause of the Rancidity of Fats. A. Mjöen. (*Forsch. Ber.*, 1897, iv., 195-203.)—The author's experiments, which were chiefly made with butter, olive-oil, and liver-oils, confirm the statements of other observers (*e.g.*, Spaeth, ANALYST, xxii., 46); but it is further shown that there is a marked difference between the oxidation changes caused in the composition of the fat by a current of air, and by the action of direct sunlight. Thus in the case of butter placed in the sun for three days the acidity number rose from 1·2 to 10·7, whilst the iodine number fell from 32·9 to 30·7; whereas the same butter, subjected to the action of a current of air alone, did not show that degree of acidity until after five weeks, and then the iodine number had fallen to 19·8. There is also a difference in the appearance of the oxidized fat, butter becoming rancid in the sunlight assuming a deep yellow colour, that

oxidized by the air keeping its colour for two or three weeks, and then becoming snow-white. The author suggests that this change may be due to the action of an enzyme similar to those which cause the discoloration of freshly-cut apples, etc., to which the name of "oxydase" has been given. Although he has proved that bacteria and moulds do not act upon fresh fat, he is not prepared to say that they play no part in a further change when once a decomposition process has commenced.

Ritsert (*Natur. Woch.*, v., 196) asserts that oxygen does not produce rancidity in fat in the absence of light; but this the author considers is conclusively disproved by his experiments. These also show that the decomposition is accelerated by raising the temperature (45° C.). C. A. M.

Japanese Wood-Oil (Tung-Oil). (*Chem. Tech. Zeit. Lack u. Farb. Ind.*; through *Deutsche Chem. Zeit.*, 1897, xii., 419. Also *Rev. Chim. Ind.*, 1897, viii., 338.) —This glyceride oil is obtained in China and Japan from the seeds of the *Aluerites cordata*, and it is being introduced into Europe as a substitute for linseed-oil in varnishes and paints. It is a clear, yellow, viscous liquid, with an odour resembling linseed-oil; when freshly prepared it is extremely poisonous; and it is used for the adulteration of gurgun balsam. Glacial acetic acid produces a turbidity at 47° C. When 5 grammes are treated with 2 c.c. of chloride of sulphur and an equal volume of carbon disulphide, the mixture solidifies on stirring for a minute and a half at ordinary temperatures; but the paste is not so hard as that yielded by linseed or castor-oil. A thin layer exposed to the air dries in about twenty-four hours to an opaque white film. If 4 grammes are heated (temperature not specified) in a 7 cm. dish, a skin forms at the edges in fifteen minutes, which in two hours becomes so tough that the contents will not fall out as the vessel is inverted; while during four hours the gain per hour is 0·36 per cent. When heated with 0·5 per cent. of litharge and a current of air passed through it, the oil acquires the consistency of a jelly in about fifteen minutes. It yields a solid elaidin. Warmed with silver nitrate for fifteen minutes, a reddish-brown colour is produced. Strong sulphuric acid converts it into a black mass; nitric acid (sp. gr. 1·4) into a solid body in two minutes, which finally becomes dark and friable. If 1 gramme of the oil is dissolved in 5 c.c. of chloroform and shaken with 5 c.c. of a saturated solution of iodine in the same liquid, a thick paste is soon produced. The constants of tung-oil are as follows:

Specific gravity at 15·5°	0·8385
Solidifying point (about)	17°
Iodine number	165·7
Saponification number	194
Specific temperature reaction	372
Insoluble fatty acids	96·4%
Unsaponifiable matter	0·44%
Free fatty acids	3·84%
Melting and solidifying point of acids	37°
Iodine number of acids	150·1

F. H. L.

[According to *Rev. Chim. Ind.* (*loc. cit.*), the free acid corresponds to 1·6 per cent. of KHO, and the sp. gr. at 30° C. is 0·9405. Deering (*Imperial Inst. J.*, 1896,

303) gives the bromine absorption as 98 per cent.: it should seem that there are at least two varieties, pale and dark; two kinds also are distinguished in China, coming from Canton and Hankow respectively. Lippert (*Zeits. Angew. Chem.*, 1897, 779) states that owing to the large amount of free acid it contains, tung-oil thickens (or "sets up") in presence of oxide pigments with inconvenient rapidity.—ABS.]

The Determination of Fat and Casein in Fæces. **H. Poole.** (*Jour. Amer. Chem. Soc.*, 1897, xix., 877-881.)—The various methods of analysing fæces have been summarized by Grausnitz (*Zeit. f. Biol.*, 1894, 328), but the author takes exception to all of them on the ground that the separations are only imperfect. Thus, the fat, which in each case is determined by extracting the dried fæces with ether and weighing the residue, contains also biliary and other products, whilst hitherto no method of separating casein from epithelium cells has been described.

Having occasion to make a number of determinations of the amount of undigested fat and casein in the fæces of children fed on milk, the author devised the following process, which he claims gives results perfectly comparable among themselves, and much nearer the truth than those of any of the older methods:

The ether extract, containing the fat, cholesterol and coloring matters from the bile, is evaporated at 100° C. and the residue dried at 110° C. It is then saponified with alcohol potash (which as a rule leaves only a trifling amount unsaponified), water is added, and the alcohol expelled by boiling. More water is added if necessary, and the solution filtered. The filtrate is extracted twice with an equal volume of ether in a separatory funnel. The liquid, thus freed from cholesterol, is evaporated nearly to dryness, water added, and the fatty acids liberated, determined in the usual way and calculated into fat.

In the determination of the casein, the fæces are extracted successively with ether, water, and alcohol, and dried, the three solvents removing most of the nitrogenous substances other than undigested casein and epithelium cells. The residue, which also contains insoluble fatty acid compounds (usually salts of iron, aluminium and calcium), is digested for several hours at 50° C. in a mixture of hydrochloric acid thirty parts, and water seventy parts, which dissolves the casein and leaves the epithelium cells and other substances. After cooling, the liquid is filtered, evaporated, and the nitrogen in the residue determined by Kjeldahl's process, and calculated to casein.

In a full analysis the amount of fatty acids in the insoluble soap would also be determined, but as these usually represent digested or partially changed fats, they did not come within the scope of the author's work, which was concerned only with the undigested portions. C. A. M.

A New Reagent for Urinary Albumins. **Bourceau.** (*C. R. Soc. de Biol.*, 1897, 317; through *Bull. de la Soc. belge*, 1897, 248.)—Urine may contain coagulable albumins or proteoses which have not the same clinical significance. Oxyphenylsulphonic acid containing a trace of salicyl-sulphonic acid will detect the albumins in the presence of the other compounds. A drop or two of the reagent added to 1 c.c.

of the urine gives an opaque white precipitate with albumins and alkali-albumins, whilst no precipitate is obtained with proteoses, peptones, alkaloids, antipyrin, salicylates, phosphates, or urates. C. A. M.

The Detection of Santonin in Urine. L. Daclin. (*Jour. Pharm. Chim.*, 1897, vi., 534.)—From 30 c.c. to 40 c.c. of the urine are treated first with lead acetate, then with crystalline sodium sulphate and filtered. The clear filtrate is divided into two parts, and evaporated in porcelain basins. The residue in one is gently warmed with one or two drops of sulphuric acid, and should santonin be present, gives an immediate violet coloration. To the other residue a few drops of alcoholic potash are added, which gives with santonin a rose colour. Or the urine may be extracted with chloroform, the solvent evaporated, and the residue tested as above. Rhubarb gives a negative result with these reagents. C. A. M.

The Determination of Phenols in Urine. J. Amam. (*Rev. Med. Suiss-romande*, xvi., 657; through *Jour. Pharm. Chim.*, 1897, vi., 361.)—The method described is a colorimetric one, based on the reaction given by phenols with Millon's reagent, and with para-diazo-benzene-sulphonic acid. Millon's reagent gives a reddish colour on warming, which however does not remain long without alteration. The other test is more characteristic and more sensitive. On adding para-diazo-benzene-sulphonic acid to phenols in alkaline solution (sodium carbonate), acid or neutral azo-benzene-phenyl sulphonates are produced, the colour of which varies from orange to red, according to the nature of the phenol. Neither of the tests can be applied to the urine itself, and the phenols must first be separated by adding sulphuric acid and distilling. The aqueous solution of diazo-benzene-sulphonic acid must be prepared immediately before use, but it has the advantage over Millon's reagent of giving the reaction in the cold, and of producing a much more permanent coloration. C. A. M.

The Analysis of Glycero-phosphates. A. Astruc. (*Journ. Pharm. Chim.*, 1898, vii., 5-8.)—For the determination of the phosphoric acid a known volume of a solution of the glycero-phosphate is neutralized with sulphuric or hydrochloric acid, with methyl orange as indicator, and the solution then titrated with a standard solution of alkali and phenol phthalein. Taking calcium glycero-phosphate as the type, the reactions are indicated by the equations:

$$2PO\begin{matrix}\diagup O \\ \diagdown O\end{matrix}\!\!>Ca + H_2SO_4 = CaSO_4 + (PO_4C_3H_7O_2)_2CaH_2$$
$$\qquad\qquad\quad \diagdown O{-}C_3H_7O_2$$

in which one molecule of sulphuric acid corresponds to two molecules of phosphoric acid,

$$\text{and } (PO_4C_3H_7O_2)_2CaH_2 + 2NaOH = (PO_4C_3H_7O_2)_2CaNa_2 + 2H_2O,$$
$$\text{or } (PO_4C_3H_7O_2)_2CaH_2 + 2NaOH = PO_4C_3H_7O_2Ca + PO_4C_3H_7O_2Na_2 + 2H_2O,$$

in which one molecule of phosphoric anhydride corresponds to two molecules of soda-

From this the quantity of phosphorus pentoxide in solution can be calculated. The results thus obtained are about 0·5 per cent. lower than those obtained by calcining a known weight of the glycero-phosphate, dissolving the ash in hydrochloric acid, and determining the phosphorus as magnesium pyrophosphate, so that the process gives very approximate figures in a very short time.

Glycero-phosphates of calcium, when dissolved, are alkaline to methyl orange, but sometimes acid and sometimes alkaline to phenolphthalein. The acid salts are almost entirely soluble without residue, whereas the alkaline salts dissolve with more difficulty and give a slightly turbid solution, but the alkalinity is always excessively feeble. Acidity to phenolphthalein indicates a certain amount of decomposition during the preparations of the calcium glycero-phosphate.

In the case of adulteration to any extent of calcium glycero-phosphate with disodium phosphate, the solution would be strongly acid to phenolphthalein, and the quantity of sulphuric acid required for neutrality with methyl orange would be far from being equivalent to the quantity of soda subsequently required for neutrality with phenolphthalein. C. A. M.

On Retamine, a New Alkaloid. Battandier and Malosse. (*Jour. Pharm. Chim.*, 1897, vi., 241, 242.)—The authors have extracted from the young leaves and the bark of the *retama sphærocarpa* by the ordinary methods of extraction an alkaloid, to which they have given the name of *retamine*. One kilogramme of the fresh plant yields about 4 grammes of the alkaloid.

Retamine crystallizes in long needles on cooling a saturated solution of it in ether or petroleum spirit, and in prismatic plates on cooling an alcoholic solution, whilst spontaneous evaporation of the alcoholic solution yields rectangular tablets. It has a very bitter taste and has no marked physiological action. It melts at 162° C., and decomposes at a higher temperature. It gives the general reactions for the alkaloids, and yields a characteristic precipitate with bismuth potassium iodide. Platinic chloride does not precipitate it. With ammonium sulphate it gives feebly the reaction of sparteine. It forms well-marked salts, which crystallize readily, and which usually contain for one molecule of retamine either one or two molecules of monobasic acid. The mean results of the elementary analysis correspond to the formula $C_{15}H_{26}N_2O$; so that retamine appears to be an oxysparteine, although different to the known artificial oxysparteines. C. A. M.

A Delicate Test for Ammonia and Organic Matter containing Ammoniacal Nitrogen. E. Riegler. (*Bul. Soc. Scientc den Bucuresci*, 1897, vi., 335; through *Chem. Zeit. Rep.*, 1897, 307.)—Para-diazo-nitraniline forms a very sensitive reagent for detecting ammonia, ammonium salts, and all nitrogenous organic compounds which evolve ammonia on treatment with strong bases (*e.g.*, albuminoids and some alkaloids). One gramme of paranitraniline is dissolved by heating with 20 c.c. of water and 2 c.c. of hydrochloric acid, the liquid is diluted and well shaken with 160 c.c. of water, and, when it is cold, 20 c.c. of a 2·5 per cent. solution of sodium nitrite are added. Ten c.c. of the liquid to be tested are treated with ten or fifteen

drops of the reagent, and 10 per cent. caustic soda solution is slowly dropped in. If ammonia is present, an orange turbidity is produced; and on agitation the liquid becomes yellow, orange, or red, according to the amount present. On acidifying with sulphuric acid the solution loses its colour, and a mass of yellow, microscopic, needle-shaped crystals collects on the surface. This body is very soluble in alcohol, and on adding a little caustic soda it gives an intense red violet coloration. The reagent becomes cloudy on keeping, but after filtration it is still serviceable.

F. H. L.

A New Process for the Determination of Crude Fibre in Food Stuffs. **J. König.** (*Zeit. für Untersuch der Nahrungs-und Genussmittel*, i., 3-16.)—This paper is the outcome of endeavours to devise a process which shall furnish crude fibre as free as possible from pentosans. The process is based upon the following results of experiments:

1. A temperature of 120° to 130° C. is not sufficient to ensure the solution of the pentosans in all vegetable substances. The most suitable temperature lies between 130° and 140° C. A higher temperature causes the hexosans to be more strongly attacked.
2. The complete solution of the pentosans by the glycerin process depends less upon the proportion of sulphuric acid added to the glycerin (within the limits of 1 to 3 per cent.) than upon the length of time allowed for the action. A 3 per cent. addition of sulphuric acid effects the solution quicker than a 1 per cent., but not proportionately to the increased quantity.
3. 200 grammes of glycerin of 1·230 specific gravity (12·5 per cent. water and 87·5 per cent. glycerin) for 3 grammes of substance appears to be the most suitable proportion, as well as the best strength.
4. The pentosans are brought into solution quicker by the employment of higher temperatures and larger proportions of acid than by the employment of lower temperatures and less acid. The most suitable length of time for boiling is on the average one hour at 131° to 133° C., reckoned from the commencement of ebullition, or half an hour at 135° to 137° C.

The process is carried out as follows: Three grammes of the air-dried substance are placed in a porcelain basin with 200 c.c. of glycerin of 1·230 specific gravity containing concentrated sulphuric acid in the proportion of 20 grammes per litre. After mixing, the basin is placed in a steam bath and subjected for one hour to a temperature of 137° C., corresponding to a pressure of three atmospheres. The lamp is then removed, and when the temperature has fallen to from 80° to 100° C., the basin is taken out, 200 to 250 c.c. of boiling water added, and the contents filtered through an asbestos filter. Should the fluid filter slowly, it must be maintained at a temperature of 80° to 90° C. throughout the filtration. A cold solution of glycerin filters badly.

The residue is washed first with 300 to 400 c.c. of boiling water, then with 50 c.c. of warm, 93 per cent. alcohol, and lastly with a warm mixture of ether and alcohol, until the washings are quite colorless. The filter is then dried and weighed, after which it is incinerated and re-weighed, the difference between the two weights giving the ash-free crude fibre.

The extraction may also be carried out at the ordinary atmospheric pressure by boiling in a flask fitted with a reflux condensor. The pentosan (furfural) determination is carried out by the phloroglucin method of Tollens. The process has been tried side by side with Henneberg's method, and the results of a number of determinations, calculated in each case upon the material in the dry state are given in the following table:

	In the original substance.		Henneberg's Process.			Pressure with Glycerin of 1·230 specific gravity and 2 p. c. Sulphuric Acid.			1·230 specific gravity and 2 per cent. Sulphuric Acid.		
	Pento-	Nitrogen.	Crude Fibre.	Pentosans in Crude Fibre.	Nitrogen Fibre.	Crude Fibre.	Pentosans in Crude Fibre.	Nitrogen Fibre.	Crude Fibre.	Pentosans in Crude Fibre.	Nitrogen Fibre.
	Per	Per cent.	Per cent.	Per cent.	Per cent.	Per cent.	Per cent.	Per cent.	Per	Per cent.	Per cent.
Rye straw	26·98	0·83	52·32	9·65	0·18	43·56 43·99 43·61 44·28	1·14 — 0·52 1·44	0·13	43·44	0·90	0·14
Pea straw	14·95	2·21	40·50	6·09	0·09	30·41	0·99	0·19	(30·41) (29·74)	0·87	0·27
Meadow hay	17·03	2·52	32·45	4·47	0·16	25·78 26·51 26·18	0·37 — 0·35	0·37	26·29	0·37	0·29
Clover hay	12·42	3·28	33·05	4·21	0·26	24·07	0·62	0·17	24·46 24·30	0·52	(0·41)
Wheat bran	17·61	3·33	10·65	0·28	0·13	11·17 11·04	0	0·20	10·68	0	0·23
Brewers' grains	19·15	4·85	16·60	1·39	0·05	15·45 15·81	0·13	0·18	16·17	0	0·13
Asparagus	10·28		17·41	2·49	0·27	14·78 14·77	0·34	0·14	15·03 15·01	trace	0·15
Cabbage	8·02	4·33	12·29	1·03	0·15	11·24 (11·89)	0·12	0·21	11·15	0	0·20
Linseed	7·91	4·57	8·93	0·78	0·22	7·63 7·85	0	0·19	7·53	0	0·13
Rye meal	—	—	0·99	0	—	1·05	0	—	1·02	0	—
Wheat meal	—	—	0·41	0	—	0·33	0	—	0·27	0	—

Fairly good results can also be obtained by heating with 200 c.c. of acidified glycerin in an open basin, only in this case it is more difficult to maintain an even temperature between 135° and 137° C. It can, however, be kept within the limits of 133° and 140° C. for half an hour, and the following results, calculated upon the dry substance, were obtained in this way:

Feeding Stuffs.	Crude Fibre.	Pentosans in the Raw Fibre.	Nitrogen in the Raw Fibre.	Feeding Stuffs.	Crude Fibre.	Pentosans in the Raw Fibre.	Nitrogen in the Raw Fibre.
	Per cent.	Per cent.	Per cent.		Per cent.	Per cent.	Per cent.
Rye straw	44·71	1·78 1·52	0·14	Wheat bran	8·51 9·09	0 0	0·09 0·11
Grass hay	25·90 25·84 25·95	0·47	0·37	Brewers' grains	13·98 13·24	0 0	0·11

The superiority of the glycerin process to the older one of Henneberg is shown very clearly in the following table, from which it appears that whereas by the Henne-

berg process considerable quantities of pentosans remain undissolved, varying with each material up to 40·73 per cent. of the pentosans originally present, by the glycerin process the quantity remaining undissolved does not exceed 6·62 per cent.

	Pentosans remaining in the Crude Fibre in percentage of the pentosans originally present.			1. Crude Fibre by Old Process.	2. Containing Pentosans.	3. Crude Fibre free from Pentosans by old Process. (1-2)	4. Crude Fibre by Treatment with Glycerin.	
	Old Process.	By steaming with Glycerin.	By boiling with Glycerin.				Steaming.	Boiling.
	Per cent.	Per cent.	Per cent.	Per cent.	Per cent.	Per cent.	Per cent.	Per cent.
Rye straw	35·77	3·82	3·33	52·32	9·65	42·67	43·86	43·44
Pea straw	40·73	6·62	5·82	40·50	6·09	34·41	30·41	30·08
Grass hay	26·24	2·11	2·17	32·45	4·44	27·98	26·16	26·29
Clover hay	33·89	4·99	4·19	33·05	4·21	28·84	24·04	24·38
Wheat bran	1·58	0	0	10·65	0·28	10·37	11·11	10·68
Brewers' grains	7·26	0·68	0	16·60	1·39	15·21	15·63	16·17
Asparagus	24·20	3·30	trace	17·41	2·49	14·92	14·78	15·03
Cabbage	12·84	1·49	0	12·29	1·03	11·26	11·57	11·15
Linseed	9·86	0	0	8·93	0·78	8·16	7·74	7·53

H. H. B. S.

INORGANIC ANALYSIS.

Volumetric Estimation of Gold. J. Petersen. (*T. Physik og Chem.*, 1897, i., 341; through *Chem. Zeit. Rep.*, 1897, 307.)—The author finds that in an acid solution gold can only be titrated accurately by means of oxalic acid or ammonium oxalate when the whole process is conducted in an atmosphere of carbon dioxide. In the presence of oxygen the finely-divided metal appears to behave like platinum-black, oxidizing the oxalic acid, and causing the volume of standard solution employed to be from 18 to 73 per cent. above the theoretical. By the use of sodium oxalate in a neutral liquid, however, perfectly satisfactory results may be obtained. The gold is dissolved in aqua regia containing a large proportion of hydrochloric acid, sodium chloride introduced, the mixture evaporated to dryness, taken up in water, and again dried down. The excess of oxalate is titrated with permanganate as usual. The chlorides of mercury and copper do not affect the process; but sufficient sodium oxalate must be added in the first instance to hold any copper oxalate in solution after the reduction of the gold. The method is not available when the gold is combined with platinum. F. H. L.

Volumetric Estimation of Antimony. J. Hanus. (*Vestnik kral. ceske spolecnosti nauk*, 1897 [II.], No. 40, 1; through *Chem. Zeit. Rep.*, 1897, 324.)—0·2 to 0·3 gramme of precipitated sulphide of antimony is filtered and washed, rinsed into a beaker, the paper added, and treated with ferric sulphate, either solid or in solution, in the proportion of 6 molecules of the latter to 1 of the former. The mixture is boiled for fifteen minutes, the following reaction taking place:

$$Sb_2S_3 + 5Fe_2(SO_4)_3 + 8H_2O = 2H_3SbO_4 + 10FeSO_4 + 5H_2SO_4 + S_3.$$

After considerable dilution, 15 c.c. of strong sulphuric acid are introduced for every 1 gramme of ferric sulphate, the liquid is cooled below 25° C., and titrated with permanganate containing 1 or 1·5 gramme per litre. The error lies between +0·3 and −0·26 per cent. of the original sulphide of antimony. F. H. L.

A New Volumetric Method for Determining Copper. W. E. Garrigues. (*Jour. Amer. Chem. Soc.*, 1897, xix., 940, 941.)—This method depends on the precipitation of the copper as cuprous thiocyanate and the alkalimetric determination of the combined thiocyanic acid. The solution is evaporated with the smallest possible amount of sulphuric acid to expel any volatile acids, is then diluted, gently warmed, and sulphurous acid added until the liquid smells perceptibly of it. The copper is precipitated with an alkaline thiocyanate, the liquid gently warmed to promote the separation of the precipitate, and the latter filtered off and washed with water.

The filter and contents are then boiled for a few minutes with a measured excess of standard caustic alkali, the liquid cooled, made up to 200 c.c., filtered, and 100 c.c. titrated with standard acid and methyl orange.

The caustic alkali converts the cuprous thiocyanate into cuprous hydroxide and alkaline thiocyanate, the latter substance being neutral to methyl orange. The reaction is in accordance with the equation:

$$2CuSCN + 2NaOH = Cu_2(OH)_2 + 2NaSCN.$$

For the separation of zinc from copper, ammonium thiocyanate must be used to precipitate the copper, and the zinc determined as phosphate in the filtrate. When potassium thiocyanate is used, the zinc precipitate is contaminated and the results valueless.

The author claims for this process greater accuracy than the iodide method is capable of, and an additional advantage is that no empirical standard is required.

C. A. M.

The Electrolytic Determination of Cadmium. D. L. Wallace and E. F. Smith. (*Jour. Amer. Chem. Soc.*, 1897, xix., 870-873).—Avery and Dales (ANALYST, xxii., 295), being unable to obtain satisfactory results by Smith's method of precipitating the metal from an acetic acid solution, the authors have made experiments to determine the original working conditions, and find that when a definite strength of current is employed and a definite surface (which, however, were not given by Smith), the method is perfectly reliable. For instance, 0·1329 gramme of cadmium oxide was dissolved in acetic acid, the solution evaporated to dryness, and the residue dissolved in 30 c.c. of water. The liquid was then heated to 50° C., and electrolyzed with a current of 0·02 ampère for 37 sq. cm. of cathode surface, the voltage was 3·5. The metal, which was completely precipitated in four hours, was crystalline and perfectly adherent. The acid liquid was siphoned off and replaced by water without interrupting the current. It was found that the time required could be diminished and good results obtained by adding 1 gramme of ammonium acetate to

the solution after the current had acted for an hour. In this particular instance the quantity of cadmium found was 0·1158 gramme, instead of 0·1162 gramme.

Experiments are also described to show that, contrary to the experience of Avery and Dales, it is possible to obtain good results by precipitating from a solution containing free sulphuric acid if the right conditions obtain. Thus, 0·1270 gramme of cadmium oxide was dissolved in 2 c.c. of sulphuric acid (specific gravity, 1·09), and the solution diluted with water to 30 c.c. It was then heated to 50° C., and electrolyzed for four and a half hours with a current of 0·08 ampère for 37 sq. cm., cathode surface, the voltage being 2·5. The deposit was crystalline and adherent, and weighed 0·1105 gramme instead of 0·1111 gramme.

With regard to the electrolytic separation of cadmium from copper, Smith showed that in the presence of free nitric acid copper was deposited, whilst the cadmium remained in solution. But Heidenreich (*Ber.*, xxix., 1,585) could not obtain satisfactory results by this process. In reply to his criticism, the authors describe experiments, of which the following is representative: To a solution containing 0·3893 gramme of copper sulphate (= 0·0988 gramme copper) and 0·1152 gramme of cadmium oxide (= 0·0985 gramme cadmium) were added 2 c.c. of nitric acid (specific gravity, 1·43). The solution was diluted to 100 c.c., heated to 50° C., and electrolyzed with a current $N.D_{100} = 0·10$ ampère. Voltage, 2·5. The metal, which was completely precipitated in three hours, was bright and satisfactory in every respect. It weighed 0·0988 gramme, and contained no cadmium. C. A. M.

The Detection of Traces of Alkaline Carbonates in the Presence of Bicarbonates or Borax. A. Leys. (*Jour. Pharm. Chim.*, 1897, vi., 440-442.)—Salts of magnesium (*e.g.*, the sulphate) distinguish between alkaline carbonates and bicarbonates, giving a precipitate with the former and not with the latter. But the author shows that this reagent, though excellent with the single salts, is not to be relied on with mixtures. Thus, a solution of a mixture containing 68 per cent. of neutral carbonate and 32 per cent. of bicarbonate of soda gave no precipitate with magnesium sulphate, and the same negative result was obtained with a mixture consisting of 60 per cent. of neutral carbonate and 40 per cent. of borax.

A saturated solution of calcium sulphate forms a much more sensitive reagent. When this is added to a solution of pure bicarbonate the liquid remains clear for some time, and then slowly begins to form microscopic crystals, but if there be only a minute trace of neutral carbonate present, an immediate white opaque precipitate is formed. The test is equally reliable in the presence of borax. C. A. M.

Acidimetric or Alkalimetric Determinations in highly-coloured Substances. F. Jean. (*Ann. de Chim. Analyt.*, vol. ii., p. 445.)—The ordinary indicators being inapplicable in the case of highly-coloured products such as ink, blacking, etc., the author recommends the following method:

If the substance give an alkaline reaction, a weighed quantity is distilled with ammonium sulphate; and the quantity of the alkali, the nature of which has been previously determined by qualitative analysis, estimated by titrating the ammonia liberated.

Acid substances should be distilled in a Schloesing apparatus with ammonium sulphate and 10 c.c. to 20 c.c. of normal alkali, the ammonia liberated by the excess of alkali being absorbed by 10 c.c. or 20 c.c. of normal acid. The excess of the latter is then titrated. C. S.

Estimation of Total Carbonic Acid in Water. S. Robertson. (*Arch. Hyg.*, 1897, xxx., 312; through *Chem. Zeit. Rep.*, 1897, 268.)—According to the volume of carbon dioxide probably present, 10 to 20 c.c. of trinormal alcoholic soda (or potash), prepared from metal and absolute alcohol, are placed in a 600 c.c. flask of hard Jena glass, a rubber stopper is inserted in the neck, and the whole weighed. About 500 c.c. of the sample are next cautiously introduced through a leading tube in order to avoid contact with the air, and the exact amount of water taken is determined by a second weighing. The liquid is then distilled till only 100 c.c. are left in the flask, the exit tube for the vapours being provided with a bulb to prevent loss of solid matter by splashing. Finally, the vessel is connected with a series of drying tubes, hydrochloric acid is run in through a tube funnel, the carbonic acid driven out by gentle warming and a current of pure air, and absorbed in potash bulbs, where it is estimated by weight as usual.

The process can also be employed for the determination of the combined carbonic acid by omitting the caustic alkali. F. H. L.

Detection of Alkalinity in Potable Waters. A. Cavalli. (*Selmi*, 1897, vii., 65; through *Chem. Zeit. Rep.*, 1897, 288.)—An extremely sensitive reagent for this purpose is found in a 1 per cent. solution of tolylene red (dimethyldiamido-tolylphenazine hydrochloride). If two or three drops of this dye are added to 50 c.c. of an ordinary alkaline water, the red colour changes to yellow.

Alessandri has suggested the addition of a few drops of red wine, the colour of which is altered under similar conditions. F. H. L.

The Determination of Oxygen dissolved in Water. G. Romyn. (*Zeit. angew. Chem.*, 1897, 658.)—The author uses a cylindrical pipette with a tap at each end and a tube above the upper tap. The pipette is completely filled with the water to be examined and the taps closed. One c.c. of a solution containing 12 grammes of manganese chloride and 8·5 grammes of potassium iodide in 100 c.c. is placed in the upper tube and allowed to enter the pipette by momentarily opening both taps. The liquids are well mixed, and then 1 c.c. of a solution of Rochelle salt (specific gravity, 1·255) and 1 c.c. of soda lye (specific gravity, 1·105) introduced in the same way.

After leaving for ten minutes, 1 c.c. of hydrochloric acid (specific gravity, 1·126) is added, the pipette emptied into a flask, and the liberated iodine titrated with standard thiosulphate. The alkaline manganese tartrate combines with the dissolved oxygen, and on the subsequent addition of the hydrochloric acid liberates from the potassium iodide an amount of iodine equivalent to the oxygen. A deduction of 4 c.c. must be made from the volume of the pipette for the reagents introduced.

The pipette may be obtained from Greiner and Friedrichs, Stützerbach.

C. A. M.

Estimation of Potash in Fertilizers. L. Ronnet. (*Rev. Chim. Analyt. Appl.*, vol. v. [21], pp. 421, 422.)—To obviate the inconvenience arising from the use of ammonium carbonate for precipitating the excess of barium in the Schloesing perchloric method of estimating potash, the author proposes to throw down the barium by means of a current of carbon dioxide.

In the case of potassium chloride fertilizers, 50 grammes are dissolved in water, made up to 1 litre and filtered; 20 c.c. of this solution are then taken and treated with 10 c.c. of a saturated solution of barium hydroxide. A current of carbon dioxide passed through the liquid for a few minutes throws down the excess of barium; and, after boiling for a few moments to drive off the residual carbon dioxide and decompose any soluble bicarbonates formed, the liquid is filtered and treated by the perchloric acid method in the ordinary manner.

For potassium sulphate fertilizers 40 c.c. (and for Kainit 30 c.c.) of baryta water will be required, and the precipitate requires more careful washing.

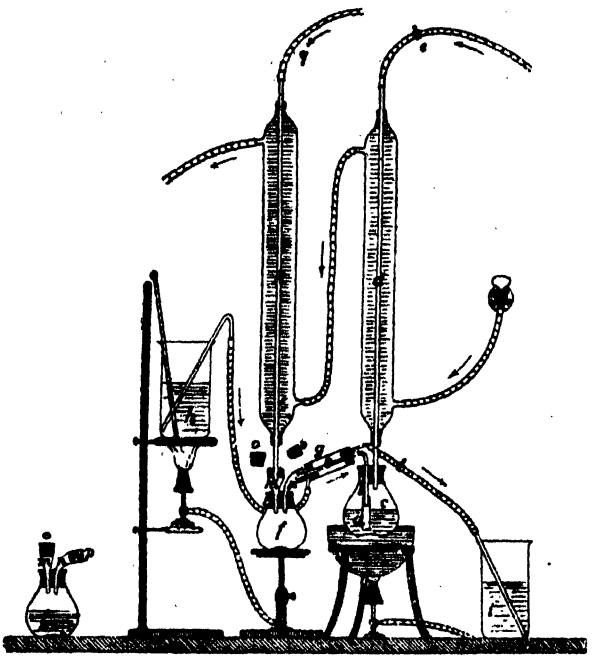
In the case of more complex fertilizers, 25 grammes are digested in hot water, and the volume made up to 500 c.c. After filtration 100 or 200 c.c.—according to the presumptive richness in potash—are evaporated to dryness in a porcelain capsule with 1 or 2 grammes of calcium hydrate, and ignited at a low temperature. The residue is taken up with small quantities of boiling water, and baryta water added so long as a precipitate continues to form. The excess of barium is thrown down, and the further treatment proceeded with as before. In all cases the liquid through which the carbon dioxide is passed should be well boiled afterwards.

C. S.

APPARATUS.

An Apparatus for determining the Solubility of Substances in Boiling Liquids. H. Göckel. (*Forsch. Ber.*, 1897., iv., 173-177.)—The usual method of determining the solubility of a substance in a boiling liquid (Fresenius, *Quant. Anal.*, ii., 794; Lassar Cohn, *Arbeitsmethoden*, 1893, 224) is to filter the boiling saturated solution through a warmed and covered funnel into a weighed flask, which is then closed with a stopper, allowed to cool, weighed, and the amount contained in an aliquot part of the liquid determined.

The author points out that in this process a considerable loss of the solvent by evaporation is inevitable, whilst the slightest cooling causes a separation of the dissolved substance on the funnel, and that these causes may lead to enormous discrepancies in the results obtained by different observers. Thus, according to Dragendorff, 1 gramme of theobromine requires for solution 422·5 grammes of boiling absolute alcohol, whereas Eminger found that 818 grammes were necessary.

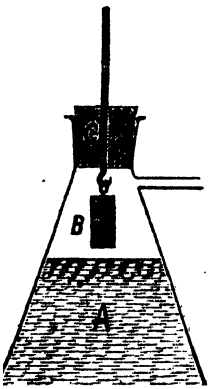


In the apparatus here described, evaporation of the solvent during the filtration is impossible, and results in good agreement can be obtained. The substance and solvent are placed in the flask, *c*, which holds about 25c. c., and in which is also placed the filter-tube, *a*, which is filled with wool. The flask is heated for about thirty minutes on the water-bath, with *d* acting as a reflux condenser. Meanwhile, the liquid in the flask is kept in motion and prevented from entering the filter, *a*, by means of gentle air-pressure supplied by bellows connected with the condenser, *k*, at *q*. After the thirty minutes' boiling, the bellows are detached from the top of *k* and connected with the condenser, *d*, by means of the tube, *e*. On now gently blowing, the boiling saturated solution passes through the filter, *a*, and tube, *b*, and falls into the flask, *f*, where loss by evaporation is prevented by the condenser, *k*. In order to prevent crystallization in the tube, *b*, the latter is surrounded by a jacket, *g*, through which passes a current of warm water heated in the beaker, *h*, to a temperature of from 1° to 2° above that of the solvent. The tube, *b*, delivers into a wider tube, *n*, to which a stopper can subsequently be fitted, and a stopper is also provided for the tube, *m*, when disconnected from the condenser, *k*. The flask, *f*, with the tubes, whose combined weight when empty is known, is weighed when containing the saturated solution, and again after the evaporation of the solvent.

The results obtained by the ordinary method (as given by Fresenius), and those with the apparatus described above, are compared in the subjoined table, in which are shown the quantities of boiling solvents required to dissolve 1 gramme of caffeine.

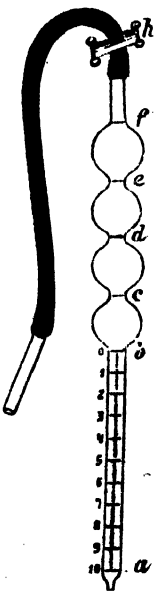
Solvent.	Ordinary Method. Grammes of Solvent for 1 gramme Caffeine.	Mean.	With Apparatus described. Grammes of Solvent for 1 gramme Caffeine.	Mean.
Ether (anhydrous) B.P. 35·5° C. ...	434·6 474·4	454·5	336·8 341·8	339·3
Acetic ether ... B.P. 72·7° C. ...	23·2 27·7	25·5	25·0 22·7	23·9
Benzene ... ... B.P. 80·4° C. ...	18·2 22·6	20·4	19·2 18·6	18·9
Chloroform ... B.P. 61·0° C. ...	5·1 5·2	5·15	6·7 6·1	6·4
Carbon tetrachloride... . . B.P. 78·1° C. ...	137·7 128·1	132·9	141·2 143·5	142·4

C. A. M.



Simple Hydrogen Apparatus. C. Aschmann. (*Chem. Zeit.*, 1897, xxi., 1049.)—This device is sufficiently explained by the diagram. A is a filter-pump flask holding about 1½ litres, two-thirds filled with dilute sulphuric acid. B is a solid block of zinc 6 cm. long and 2 cm. in diameter, cast round a copper wire with a loop at the top, by means of which it is suspended, as shown. The cylinder is pushed into or drawn out of the acid, according as a current of gas is required or not.

F. H. L.

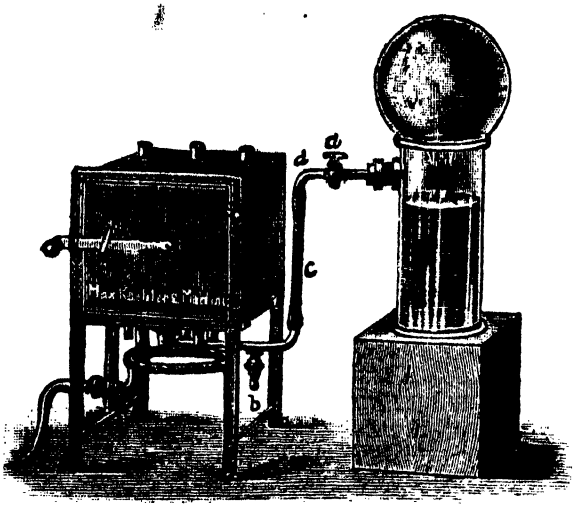


A New Measuring Pipette. Otto Bleier. (*Chem. Zeit.*, 1897, 1028.)—By means of the comparatively short pipette, illustrated in the accompanying cut, any quantity of liquid up to 50 c.c. can be accurately measured. The instrument is divided into five sections, each of which has a capacity of 10 c.c., the lowermost being graduated in tenths of 1 c.c. The amount of liquid delivered is regulated by the screw clamp *h*. The constrictions between the bulbs have the same diameter as the graduated portion.

W. J. S.

Drying Oven for Maintaining Constant Temperatures. M. C. Schuyten. (*Chem. Zeit.*, 1897, xxi., 1049.)—This consists of a constant level apparatus placed in connection with an ordinary copper water-oven. When a temperature of 97·5° C. is required, both parts contain water, and the whole is worked in the usual way. But if a higher temperature (98° to 105°) is desired, the oven is filled with a strong solution of sodium nitrate, while the reservoir is charged with plain water as before. The wooden block is of such height that equi-

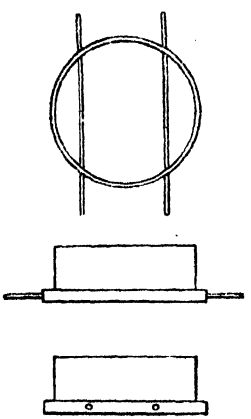
librium obtains when the drying-oven is practically full of liquid; the cock *a* is closed, the burner lighted, and as soon as ebullition sets in, *a* is opened. If a thermometer shows that the temperature is too low, more solid sodium nitrate is introduced through one of the openings in the oven; but if it is too high, *a* is shut, and some of the solution is drawn off from *b*. After a few trials, the proper concentration will be obtained; and then the temperature will remain steady, even if the gas-flame varies in size, so long as the liquid continues to boil; for the loss by evaporation is constantly made good by the addition of pure water.



Sodium nitrate seems best adapted for this purpose because it does not act appreciably on copper; but it must be very pure, and especially free from chlorides or sulphates.

F. H. L.

New Electrolytic Diaphragm. H. Pauli and L. Pincussohn. (*Chem. Zeit.*, 1897, xxi., 1048.)—The ordinary commercial porous pot is inconvenient for electrolytic analysis, as it is difficult to watch the process going on inside, and the outer electrode must be bent into a circular form. The improved cell recommended by the authors is made much shorter and wider, as will be seen in the accompanying illustrations. At its upper end it is strengthened by a flange in which four holes are bored; two wires are passed through them, and in this manner the cell is suspended within the outer vessel. The latter need not be a beaker; a flat crystallizing-pan is preferable. The electrodes are therefore not curved, but straight and parallel; and thus it is easier to maintain a uniform current density. The apparatus has been successfully employed in carrying out Weyl's process for the estimation of carbon in hard steels. F. H. L.



MISCELLANEOUS.

We are pleased to hear that the governing body of University College, Nottingham, has conferred the title of Emeritus Professor on Dr. Clowes on his retirement from the Professorship of Chemistry in the College to undertake the duties of Chemist to the London County Council.

THE ANALYST.

MARCH, 1898.

PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS.

THE annual meeting was held on Wednesday, February 2, 1898, in the Chemical Society's Rooms, Burlington House, the President (Dr. Bernard Dyer) occupying the chair.

The minutes of the previous meeting were read and confirmed.

The following gentlemen were proposed for election. As members: H. H. Dains, F.I.C., 3, Cantonment, Vizianagram, India; A. D. Hall, M.A., Principal of and Professor of Chemistry at the South-Eastern Agricultural College, Wye, near Ashford, Kent; Otto Rosenheim, Ph.D., 57, Chancery Lane, London; P. Schidrowitz, Ph.D., 57, Chancery Lane, London. As associates: C. A. Hackman, assistant to Mr. A. C. Chapman; and A. P. Davson, assistant to Mr. Bodmer. Mr. L. K. Boseley was elected a member of the Society, and Mr. F. R. O'Shaughnessy as associate.

The HON. TREASURER (Mr. E. W. Voelcker), in presenting his report and accounts for the past year, drew attention to the fact that, while the amount of the year's expenditure was larger than in the case of the previous year, the balance actually standing to the Society's credit was larger. He was glad to be able to remark that the subscription list was in a more satisfactory condition than it had been in since 1895, there being, as a matter of fact, only five subscriptions remaining in arrear; and he desired to express his appreciation of the kindness with which the members generally had responded to his applications. The chief items in the Society's expenditure were those connected with the ANALYST, and these items were larger than in the previous year, this being in great measure due to the cost of the General Index which had been published during the year just concluded, and of which the cost of printing alone had been £53. He might observe, however, that the items referring to the ANALYST constituted the only portion of the accounts which showed an increased expenditure over the preceding year, the Society having, in fact, been as economical as possible in every direction, excepting that which, he ventured to say, was the one in which they were right in being lavish in their expenditure. Any surplus money they had to spend, he thought they ought to spend in raising that journal to as high a standard as possible.

Mr. JOHN H. B. JENKINS moved the adoption of the report and accounts as

presented by the Hon. Treasurer, at the same time proposing a hearty vote of thanks to the Hon. Treasurer and to the Hon. Auditors (Mr. John Hughes and Mr. Bertram Blount).

Mr. EKINS seconded the motion, which was unanimously carried.

The HON. TREASURER and Mr. HUGHES responded, the latter saying that the accounts were kept with great accuracy of detail, and in such a clear and admirable manner that the task of auditing them was a very light one.

The President then delivered the following address.

ANNUAL ADDRESS OF THE PRESIDENT.

I REGRET to have to begin the remarks I have to make by chronicling the fact that we have lost by death during the past year four of our members. The first name among them is that of our honoured honorary member, Fresenius. His loss has been deplored all over the chemical world; but there is no chemical body or society to whom it comes home with such force as it does to a society which, like our own, is devoted specially to the study and advancement of analysis, the branch of chemical work to which Fresenius devoted his long and fruitful life. His well-known handbooks are among the earliest and best-used possessions of the student, and they are, at the same time, among the most treasured possessions of the most mature analyst among us. Our past-President, Mr. Hehner, who was privileged to be his pupil, has already recorded, much more ably and eloquently than I could, in the pages of our journal the appreciation necessarily felt by all analysts of the work of Fresenius and our sorrow at his death; but I nevertheless feel that it would not be becoming to let our annual meeting go by without once again recording our sense of the loss we have all sustained.

Mr. F. M. Rimmington, whose loss has also been chronicled in the ANALYST, was one of the oldest members of our Society, and, I think, one of the oldest public analysts in the country; and, although advancing years prevented his taking a very prominent part in the affairs of our Society for some time past, he was formerly active in analytical research, more especially in connection with the examination of drugs.

Dr. G. A. Rogers was also an old member of the Society, and an old public analyst, although he had not, I think, for many years taken any active part in our society.

Mr. C. H. R. Moore was for a number of years a member of the Society, and for several years previously an associate. He was originally one of my own pupils and assistants, and one for whom I had a deep personal attachment, and his early death has been, as I know, a subject of grief to a number of our members who knew him as a familiar friend before he left England. At the time of his death he was working at agricultural chemistry in Jamaica.

We have lost during the year by resignation two of our ordinary members, while the names of three members and one associate have fallen out of our list by reason of efflux of time since their last communication with our treasurer.

We have thus lost during the year one honorary member, eight ordinary members, and one associate. We have had, however, the pleasure of adding to our list of honorary members the name of Dr. W. J. Russell, F.R.S., who has earned the thanks of the whole profession for the able way in which for three years he filled the presidential chair of the Institute of Chemistry. Our appreciation of his services in this capacity cannot be better expressed than by saying that he proved himself a worthy successor to Dr. Tilden, with whose name will always be associated what I venture to call the rejuvenescence of the Institute of Chemistry; and we have recorded our sense of Dr. Russell's services to our profession in the same way as that in which we recorded our recognition of those of his predecessor, Dr. Tilden.

This would perhaps be a fitting place to congratulate both our own Society and the Institute of Chemistry upon the fact that Dr. Russell, in resigning his office, has been succeeded by a President no less worthy than our own immediate past-President, Dr. Stevenson, whose hard work in the chair of this Society will be long remembered by all those of us who had to work with him in an official capacity. Under his presidency, we hope to see the Institute holding, in addition to its other examinations, an examination in the much-vexed subject of pharmacology and therapeutics, so as to enable candidates for public analystships to produce evidence of the supplementary qualification in this subject required by the Local Government Board, and thus removing a difficulty frequently experienced. We as chemists have long striven to impress upon the Local Government Board the urgent necessity of a strong chemical qualification for public analysts; but from the unfortunate frequency with which in the past the Local Government Board has expressed itself satisfied with the qualifications of candidates possessed of medical diplomas, but possessed of very slender chemical qualifications, it would almost appear as though in the eyes of the Local Government Board a therapeutical qualification had sometimes weighed even more heavily than a chemical one. If the Institute of Chemistry is able to see its way, as we now hope that it will, to supplement its already sound and thorough chemical examinations by the pharmacological and therapeutical examination already referred to, it will form a completely qualifying body for the special department of the profession which this Society primarily represents.

At one time, in view of the absence anywhere of any examination sufficiently comprehensive and at the same time sufficiently special to test the qualifications of candidates for public analystships, there was on the part of many members of our own Society a wish that we—the Society of Public Analysts—should examine, and grant a diploma. Others of us, however, felt that to further increase the number of examining bodies was exceedingly undesirable, and that this important work was one best reserved for the Institute of Chemistry, which, by its objects, its charter, and its organization, was eminently fitted for carrying out this, among other special as well as general examinations; and it is satisfactory now to know that such an end to the discussion seems likely to be happily brought about.

We have elected during the year ten new ordinary members, while three associates have been transferred to the rank of members. One member who had formally resigned has cancelled his resignation, and four new associates have been added to our list.

The strength of the Society, as compared with its strength at the close of the two preceding years, is as follows:

	January, 1896.	January, 1897.	January, 1898.
Honorary members	10	11	11
Members	210	218	223
Associates	26	29	31
Total	246	258	265

It will be seen that during the past year we have on the whole gained five members and two associates, as compared with a gain of eight members and three associates in the preceding year. Although, however, the Society will be seen to be growing, the growth is too small to be looked upon as satisfactory, having regard to the large enterprise we have held in hand since we enlarged the scope of our journal. I would ask our members to bear in mind that, although our title is a restricted one, our membership is open to any chemist who has the responsible conduct of an analytical laboratory, and I sincerely hope that any member of the Society who has friends who are eligible for our membership, and who are still outside our ranks, will at once impress upon them that to support the only journal in the English language devoted to a comprehensive record of the progress of analytical chemistry is the duty of every member of the profession; and that that support is best given by joining our Society.

While, however, I am very earnest in this appeal, I do not wish it to go out as a presidential utterance that we are in actual or immediate need of funds. During our earlier and less ambitious years we accumulated, for a young Society, a not inconsiderable amount of funded property. During the year that has just passed we have not had to trench upon our investments, but I regret to say that we have not been able to add to them. From the treasurer's statement you will see, indeed, that our income has increased, but you will see as well that our expenses have also increased materially, and it is scarcely necessary to remind you that the great bulk of our income is expended on our journal.

Of the activity of our Editorial Committee, and more especially of our able and energetic Editor, Dr. Sykes, I need scarcely remind you; but I should like to emphasize our work statistically by pointing out that during the year, in addition to 36 original papers, we have published in the pages of the ANALYST no less than 283 abstracts. My predecessor, Dr. Stevenson, was last year at the pains of tabulating the abstracts which we published during 1896, and in order to show the growth of this work I have made a similar analysis of the abstracts published during 1897, which I now give side by side with his.

	Abstracts published in the ANALYST. 1896.	1897.
Food and drug analysis	55	86
Toxicological analysis	9	5
Organic	86	87
Inorganic	86	91
Apparatus	9	14
Total	245	283

It will be seen, therefore, that our abstracts were 38 more than in the previous year. We endeavour, as far as possible, to abstract every paper of analytical importance published in any country and in any language, and we are anxious—I speak for the moment on behalf of the Editorial Committee—to make our journal still worthier of the Society; and in order to do this we want two things. As I have already said, we want an enlargement of the membership of the Society, and we want an increase in the number of original papers read before the Society. During the past year 34 papers have been read and discussed at our monthly meetings, as against 29 during the previous year; but I should like to see a much larger record than this, and I should like to see a wider range of analytical subjects. I am afraid that many chemists read or publish elsewhere papers that ought properly to be read and discussed at our meetings, on account of our name, which leads them to suppose that the only papers acceptable here are those having to do with subjects falling within the statutory duties of public analysts as such. There cannot be a greater mistake. Even those among us who are primarily public analysts, are, for the most part, in general practice, and are interested, individually or collectively, in the widest possible range of analytical subjects, and any paper on any subject whatsoever connected with analytical chemistry is heartily welcomed at the meetings of our Society. I say this, not for the information of those who are present, but for the sake of others who are not members of our Society, under whose eyes this address may fall, for I am glad to say that the ANALYST has a large chemical reading public outside of our Society, and it is in the ranks of that chemical reading public that I would fain find more contributors of papers and more recruits for the Society itself.

I do not wish, however, on behalf of the Council and officers of the Society, to appear to be in any way ungrateful for the 34 papers read before us during the year, many of which have been most interesting and most valuable to us, and of which the following is a list:

"Note on weighing out Fats." By Charles E. Cassal.
"A Specific Gravity Pipette." By W. F. Keating Stock.
"A modified Schmidt Process." By R. W. Woosnam.
"Some Analyses of Water from an Oyster Fishery." By Charles E. Cassal.
"The Composition of Meat-extracts and similar Products." By Otto Hehner.
"Remarks on Formaldehyde." By Charles E. Cassal.
"The Distillation of Formaldehyde from Aqueous Solution." By Norman Leonard, B.Sc., Harry M. Smith, and H. Droop Richmond.
"The Estimation of Milk-sugar in Milk." By H. Droop Richmond and L. K. Boseley.
"The Constitution of Milk." By H. Droop Richmond.
"Copper in Preserved Vegetables." By R. Bodmer and C. G. Moor, M.A.
"Coffee Palace Coffee Infusions." By E. G. Clayton.
"The Detection of Mixtures of Diluted, Condensed, or Sterilized Milk with Fresh Milk." By H. Droop Richmond and L. K. Boseley.
"The Separation and Identification of the Typhoid and Colon Bacilli." By F. Wallis Stoddart.

"Notes on Alcohol." By J. F. Liverseege.

"The Value of the Nitrogen Factor in the Analysis of Decomposed Milks." By Alfred Smetham.

"Notes on the Influence of Boric Acid upon the Action of Digestive Ferments." By R. A. Cripps.

"Note on Heavy Metals in Cheese." By Alfred H. Allen and F. Hudson Cox.

"On the Structure of the Fat Globules in Cows' Milk." By Professor V. Storch, of Copenhagen (communicated by H. Faber).

"On Tabarie's Method for the Estimation of Alcohol." By Norman Leonard, B.Sc., and Harry M. Smith.

"The Determination of Soluble Proteids by Chlorine Precipitation." By S. Rideal, D.Sc., and C. J. Stewart.

"Note on the Bacteriolysis of Gelatin." By S. Rideal, D.Sc., and R. Orchard.

"Note on Commercial Ground Cassia." By W. F. Keating Stock.

"An Improved Method of Determining Proteid and Gelatinoid Substances." By Alfred H. Allen and A. B. Searle.

"The Estimation of Acetates in the Presence of Inorganic Salts." By Bertram Blount.

"The Estimation of Carbonic Acid in Natural Waters." By Clarence A. Seyler, B.Sc.

"Note on the Graduation of Leffmann-Beam Bottles." By G. E. Scott-Smith and A. B. Searle.

"The Detection of Gelatin in Cream." By Alfred W. Stokes.

"A new Milk Preservative." By Alfred W. Stokes.

"A new Milk Adulterant." By Alfred W. Stokes.

"An improved Milk Scale." By H. Droop Richmond.

"Milk Analysis and Certification." By James Edmunds, M.D.

"A Butter eighteen years old." By E. G. Clayton.

"A Method of Estimating Tannic Acid by means of the Polarimeter." By R. F. Wood-Smith.

"Note on Mineral Matter in Commercial Bleached Ginger." By E. J. Bevan.

As all, or nearly all, of these papers have been actually published in the ANALYST, I need not now linger to review them, but I should like to refer especially to two among them. The first was a paper "On the Separation and Identification of the Typhoid and Colon Bacilli," by Mr. Stoddart. It is gratifying to us as analysts that this, one of the most interesting papers published upon a most important and interesting topic, should have been the work of one of our own members, and a "working public analyst." I am convinced that those of us who undertake the analysis of water—and what analyst does not?—will ultimately have one and all to adopt bacteriological methods as supplementary to our chemical analyses, if we are determined to place ourselves in possession of the fullest possible evidence to be obtained from the samples of water submitted to us. But it must be recognised that bacteriological science is not the all-powerful thing that the unscientifically educated public is apt for the time being to suppose. Its difficulties have nowhere been better demonstrated than by Mr. Stoddart in this room, when he showed us how complex

was even the single group of organisms usually associated together under the common designation of the *Bacillus coli communis* group. The separation and identification of the typhoid organism by the really beautiful methods demonstrated by Mr. Stoddart and its differentiation from its innocent cousins, is no doubt under some circumstances possible, but it appears to be very doubtful whether these circumstances are the ordinary everyday circumstances under which the water analyst is called upon to act. What has occurred with reference to the terrible epidemic at Maidstone will act as a wholesome check to those who have cried out that bacteriology is everything, and that the bacteriologist is destined to supersede the chemical water analyst. In Maidstone we have had one of the most severe epidemics of modern times, clearly traced, as far as circumstantial evidence can possibly go, to a particular portion of the public water-supply. Our past-President, Dr. Adams, as Medical Officer and Public Analyst for Maidstone, was charged with the duty of investigating this source of supply, and his analyses have given clear indications of undesirable contamination. But Dr. Adams has had associated with him in the inquiry one of our ablest bacteriologists, Dr. Washbourn, of Guy's Hospital, who had before him what, from the bacteriologist's point of view, must be regarded as a magnificent opportunity for demonstrating the value of bacteriology as a means of indicating specific infection. What has been the result? Dr. Washbourn, after a most exhaustive examination, has been unable to separate or identify the typhoid bacillus in the very water which, with more than mere presumption, may be said to have been distributing the disease-germs which he sought to find. Not only has Dr. Washbourn been unable to detect the typhoid bacillus, but he expresses his doubts as to whether in any of the earlier cases in which it is said to have been identified in connection with the dissemination of disease by water, the bacillus has really been discovered, its reputed discovery having been made when bacteriological methods were less precise than they are now, and when, I may add, it was easier to jump to rash conclusions. In fact, although the typhoid bacillus may now be capable of satisfactory identification when once we can grow a colony of it, the difficulty of growing, or rather of lighting upon a colony, among the overwhelming progeny of concomitant organisms of the *Coli communis* group is so remote as to be, as Dr. Washbourn seems to recognise, almost outside the field of practical laboratory work.

But Dr. Washbourn's bacteriological examination must not, however, on this account be said to have been a failure. He has shown that in many cases the bacteriological condition of the water, while it has not sufficed to demonstrate specific infection, has sufficed to demonstrate fæcal pollution in certain sources of the water-supply, thus giving independent confirmation of the chemical results of Dr. Adams.

I will not say more about Maidstone, because I have asked Dr. Adams to come here and tell us, from the analytical point of view, the story of his work in connection with the epidemic, and I hope that at an early meeting he may be able to do so. But before leaving the subject, I would again say that, although I look forward with very little confidence to bacteriology as a practical means of identifying the typhoid bacillus under the ordinary circumstances of the spread of the disease by polluted water, I must repeat that I have the greatest possible faith in the value of bacteriology

as an adjunct to the chemical analysis of water. For some years now I have made bacteriological cultures in every water sent to me for analysis, ascertaining the number of colonies per volume capable of growth on gelatin at the ordinary temperature, the number of colonies growable on agar at blood heat, and the number growable on acidified phenolized agar at the same temperature. Any analyst who will systematically do this side by side with his chemical analysis, and steadily compare the results, will after a time feel himself very greatly strengthened in pronouncing his opinion on the great majority of cases that come before him. Occasionally anomalous cases occur, and it is obvious that the origin of a water greatly affects the opinion to be arrived at; but the same thing is true of many of the chemical features of a water analysis. I think that any water analyst who has once systematically habituated himself to make such a bacteriological examination as that indicated would feel his judgment sadly lamed if he had for any reason to give up the practice, and to fall back upon his chemical results alone. I think, however, that the idea that the practice of such elementary bacteriology as is needed for practical purposes in the analysis of water requires for its understanding or accomplishment medical training or medical experience, or that the medical bacteriologist can find out something about a water which is beyond the powers of the ordinary public analyst who has learned simple bacteriological manipulation, has received its death-blow at Maidstone. Bacteriological examination of drinking-water is destined to become as everyday an operation as the determination of chlorides; but it is an operation that we can perfectly well undertake ourselves, and if we would recognise this, and learn to do it, our County Councils and local authorities will not think it necessary to hand over such work, which should properly be ours, to our medical brethren.

The other paper that I would single out for reference is that of Professor V. Storch, communicated to us last summer by our member, Mr. Harald Faber. I refer to this paper because it claims—and I do not think the claim, although disputed, has been set aside—to settle the long-debated question as to whether the fat-globules in milk are or are not surrounded by a special pellicle. Professor Storch, after a great deal of work, concludes that the pellicle after all exists, and, furthermore, that it consists of a special and hitherto unrecognised proteid body.

Among what may be called the domestic incidents in our Society, we have had to chronicle during the year a very pleasant country meeting, held at Derby, under the hospitable auspices of Mr. Archbutt and Mr. White, when we had the pleasure of visiting the Midland Railway Works, the Crown Derby Porcelain Works, and the very interesting printing and lithographic works of Messrs. Bemrose and Sons.

My predecessor, speaking from this chair a year ago, concluded his address with a devout hope that the Government would during the year introduce into Parliament a Bill based on the report of the Select Committee of the House of Commons on Food Products Adulteration, and that that Bill might not be crowded out by the plethora of less important matters. A part of that hope—viz., the introduction into Parliament of a Bill—was realized. You know already, however, that no Bill was introduced till the very end of the session, and then only as a mere formality; and you know also how fragmentary and incomplete was the Bill which the President

and Secretary of the Local Government Board then produced. This Bill has been the subject of very anxious and careful deliberation on the part of your Council, and as a result of that deliberation a report has been issued, fully pointing out the directions in which the Bill should be amended in order to give substantial effect to the recommendations of the Select Committee. This report has been placed before the Local Government Board, and is being issued to all public authorities interested in the carrying out of the Food Acts, as well as to all members of Parliament. As a copy of the report has been placed in the hands of every member of the Society, and as it has been fully published in the ANALYST, and as, furthermore, the subject was discussed at some length by Mr. Kearley, M.P., at our recent annual dinner, I need not take up your time now by discussing it further. Suffice it to say that we were all severely disappointed by the Government Bill; and that we hope that after all it may not have been the final intention of the Government to stop short at the very insufficient alterations in the law therein proposed, but that we may in the coming session be gladdened by the production of a fresh Bill, which shall, as I have said, substantially give effect to the recommendations of the Select Committee of the House of Commons, more especially with regard to the appointment of that Standing Committee of Reference, which is indispensable for dealing with the many vexed questions calling for settlement which cannot themselves be dealt with in a single Act of Parliament, however comprehensive.

In concluding these somewhat desultory observations, I have, as your President, to acknowledge the heavy and untiring work performed by our officers—the honorary secretaries and the honorary treasurer. The year has been, owing to the important matters which we have had to consider, a toilsome one for our secretaries, and I am afraid that I cannot promise them much rest in the year that is to come; but I am sure their hearts are so fully in the multifarious work we shall have to get through, that the harder it becomes the more satisfied they will be.

Mr. JOHN HUGHES, in proposing a vote of thanks to the President for his address, desired to make one remark on the subject of the extension of the Society. The President thought, and rightly, that they should look to an increased number of members as a means of increased revenue; and, while agreeing in this, he would venture to suggest that as the journal became more valuable, its price should be increased to those outside of the Society. He thought that if it were made more expensive to obtain the journal without joining the Society, the result would be an increase in membership. As it was, many persons obtained the advantages of the journal without being members of the Society. With regard to what the President had said with reference to the Institute of Chemistry in his address, he (Mr. Hughes) was not inclined to agree with the resolution which had been passed by the Institute Council, but he had not been able to be present at the last Council meeting. He did not see why the Institute should be called upon to furnish authority as to qualification in therapeutics; he thought that what the Institute had to do was to give qualification in chemistry, and he did not see how it could give to chemists a qualification which many of its members themselves did not possess, or who the examiners were to be. He would have much preferred that the action should have been left to some other society—at all events, for the present; but he had no doubt that the Council of the

Institute had gone into the question very much more fully than he himself had been able to do, and that they had been enabled to come to a right conclusion. Their own President had himself held at first somewhat different views on the question, and other members also had altered their views upon considering and inquiring into the matter, and he had no doubt that there were very good reasons for the passing of the resolution. Referring again to the accounts, he certainly thought that the sums derived from life membership subscriptions should be funded in future, because they represented compositions of possible future payments.

The motion was seconded by Mr. PERCY A. E. RICHARDS, and carried unanimously.

The PRESIDENT said that he was pleased that Mr. Hughes thought that possibly, after all, the Council of the Institute of Chemistry had come to a correct conclusion in the matter of therapeutical qualification. He certainly thought that the best thing was that all necessary examinations should be held by the Institute of Chemistry, and there would surely be no difficulty in finding proper examiners in the new subject. That, however, was not a meeting of the Institute of Chemistry, but of the Society of Public Analysts; but the matter was so important a one, as affecting the particular branch of the profession in which so many members of the Society were engaged, that he had thought it right to take that opportunity of drawing attention to it. He now had to propose a vote of thanks to the President and Council of the Chemical Society for allowing the Society for another year to have the use of their rooms. If there were not some other society who would open its doors in this manner to them, the Society of Public Analysts would not be in such a good financial position as it then was, for it would be taxed with heavy expenses for rental. He therefore had pleasure in moving from the chair that the cordial thanks of the Society of Public Analysts should be given to the President and Council of the Chemical Society for the use of their rooms during the past year.

The motion was carried unanimously.

The following papers were then read:

"Copper 'pure for analysis.'" By J. W. Westmoreland.

"Note on the tests for distinguishing boiled from unboiled milk." By H. Leffmann, M.D.

The following is a list of the officers and Council elected for 1898:

President.—Bernard Dyer, D.Sc.

Vice-Presidents (who have filled the office of President).—M. A. Adams, F.R.C.S.; A. H. Allen; Sir Chas. A. Cameron, M.D., F.R.C.S.; A. Dupré, Ph.D., F.R.S.; Otto Hehner; Alfred Hill, M.D., F.R.S.E.; J. Muter, M.A., Ph.D., F.R.S.E.; Thos. Stevenson, M.D., F.R.C.P.

Vice-Presidents (who have not filled the office of President).—A. P. Aitken, D.Sc., F.R.S.E.; W. W. Fisher, M.A.; J. A. R. Newlands.

Hon. Treasurer.—E. W. Voelcker, A.R.S.M.

Hon. Secretaries.—E. J. Bevan; Charles E. Cassal.

Other Members of Council.—Bertram Blount, A. Wynter Blyth, M.R.C.S., R. Bodmer, A. C. Chapman, Sidney Harvey, C. G. Moor, M.A., S. Rideal, D.Sc., J. E. Stead, F. Wallis Stoddart, Walter J. Sykes, M.D., J. A. Voelcker, M.A., B.Sc., Ph.D., John White, W. C. Young.

THE ANNUAL DINNER OF THE SOCIETY OF PUBLIC ANALYSTS.

THIS was held on Tuesday evening, January 18, at the Criterion, under the chairmanship of the President, Dr. Bernard Dyer. About sixty-five members and guests were present, amongst the latter being Mr. Hudson E. Kearley, M.P., Dr. Thos. Stevenson (President of the Institute of Chemistry), Professor Frank Clowes, D.Sc. (President of the Society of Chemical Industry), Dr. Seaton (President of the Society of Medical Officers of Health), Mr. Walter Hills (President of the Pharmaceutical Society), Professor W. A. Tilden, D.Sc., F.R.S., Professor Armstrong, F.R.S., Mr. C. E. Groves, F.R.S., Professor Dunstan, F.R.S., Dr. Pakes, Dr. Messel, Mr. Fred. W. Beck, Mr. Aubrey W. Rake, Mr. G. C. Barnes, Mr. H. Owen, and Mr. J. F. Owen. The Chairman of the London County Council (Dr. W. J. Collins), who was to have proposed the toast of "The Society," was prevented from being present owing to the funeral of his colleague, Mr. Ashley Ponsonby.

The toast of "The Queen" having been duly honoured,

The CHAIRMAN proposed "The Houses of Parliament." He said that when he had the honour, a year ago, of proposing the same toast, he was buoyed up with the hope, as most of them were, that before his year of office had expired Her Majesty's Government would have produced a Bill which would have enabled Parliament to earn their thanks and congratulations for having placed upon the Statute Book an Act substantially giving effect to the recommendations of the Select Committee of the House of Commons on Food Products Adulteration. That hope, as far as the past year at all events was concerned, was doomed to disappointment. The first shock was received when, at the opening of Parliament, they read the report of the Queen's Speech, and saw with sorrow that there was no mention in that Speech of new food legislation. Their good friend Mr. Kearley (applause) made a gallant attempt to stimulate the Government into action by himself producing a very excellent Bill (hear, hear), a Bill which might very well, with perhaps a few alterations, have been adopted by Her Majesty's Government with a considerable saving of time and trouble to that Department, which Mr. Chaplin had lately told his constituents was so sadly overworked. While Mr. Kearley's stimulant was not altogether without effect, it had, however, to act on a constitution that was not very acutely responsive to stimulants, and its action was feeble. It resulted in the production, by the President of the Local Government Board, at the end of the session, of a short Bill which was exceedingly disappointing (hear, hear). The saddest feature, perhaps, about that Bill was the signature on the back of it of Mr. T. W. Russell, the Parliamentary Secretary of the Local Government Board, who had sat as Chairman of the Food Products Adulteration Committee, and who with his own hand had drafted most of the recommendations of which the very Bill of which he was obliged to stand sponsor took no notice. There was only one ray of hope in connection with the whole matter, and that was the statement made by Mr. Chaplin when he withdrew the Bill simultaneously with its introduction, a statement that he invited expressions of public opinion on the matter. It was to be hoped that even by that time public opinion had been expressed sufficiently strongly to assure the

President of the Local Government Board that the small measure which he introduced satisfied not a single one of the many interests affected by food legislation. The Bill, such as it was, had been so recently dissected and discussed by the Council of the Society of Public Analysts, whose report had been circulated among the members of the Society, that he did not propose then to refer to any of its details. The report of the Select Committee was throughout pitched in a very strong and very clear key, and its dominant note all through was the necessity for the appointment of a Standing Departmental Committee, or Court of Reference, which should deal with matters which could not possibly be dealt with completely in one comprehensive Act of Parliament. Public Analysts, as executive officers under the Sale of Food and Drugs Act, knew perhaps better than most people how urgent was the necessity for the formation of such a committee, if new legislation was to have any real effect for good on the food-supply of the country. The question, although a Parliamentary one, was not in any sense a party question. They were on the eve of a new session, and were hoping that Mr. Chaplin might introduce a Bill which should at any rate be some improvement on the one which he introduced a few months ago, and they hoped and trusted, and knew, that when that Bill was introduced their friend Mr. Kearley and those who thought with him in the House of Commons, no matter whether they sat on the one side of the House or on the other, would bind themselves firmly together to meet the necessities of the case, and to see that that Bill, if it required amendment, should be satisfactorily amended; and they hoped that the efforts of those friends would finally result in securing a satisfactory Act, which for many years to come should put an end to the many points on which so much burning discussion had arisen within the last few years. Mr. Kearley not only represented an important constituency of the British public, but he also, in his own person, represented the great mercantile interests of this country, and he was able to deal with this matter with a knowledge which was denied to many of those who sat with him in the House of Commons. Mr. Kearley had thrown himself for many years past with very great energy into this question, and they as Public Analysts, who wanted to do their duty better than they were able to do it at present, were grateful to him for the help that he had given to them in the past, and would give to them in the future; and he (the Chairman) would ask them, in drinking to the Houses of Parliament, to drink with that toast especially the health of their good friend and honorary member of the Society, Mr. Kearley (applause).

Mr. Hudson E. Kearley, M.P., said that he felt it an honour to have his name associated with that toast. The Chairman, in introducing it, had struck a note which appealed to him at once, in saying that at all events there was one question which had no party bearing, and that was the great question in which Public Analysts were so much interested. He was glad to be able to subscribe to that, and to go further and say that, in connection with the life of a member of Parliament, there was plenty of work to be done outside the limits of pure party obligations (hear, hear); and if there were pleasures that fell to the lot of members of Parliament, he would say that the most cherished were those that came from taking part in work that at all events contributed something towards the public advantage and welfare, and was not limited and confined as party work pure and simple naturally was. He

was sure that the company on that occasion were more interested in this particular question of food-products adulteration than in any other question, and therefore he would trespass on their attention by making some observations as to the course which legislation attempted to take during the last session, and as to the prospects that it might have during the forthcoming one. The Chairman had referred to the considerable disappointment experienced on all hands by the inadequacy of the legislation proposed in Mr. Chaplin's Bill. That Bill was a remarkable one, in so far as it was introduced at the expiry of the session merely in fulfilment of a pledge that a Bill should be introduced. No one within the walls of Parliament thought for a single moment that the Bill was intended seriously. Mr. Chaplin himself, indeed, rather described it as a *ballon en l'air*, and evidently expected that during the Parliamentary recess his abortive Bill would be torn to pieces, with the object of inducing him to introduce a more satisfactory measure. There would be plenty of criticism as to the direction which Mr. Chaplin should take in the coming session in bringing about a more perfect measure. The Bill introduced at the close of the last session simply, in his (Mr. Kearley's) opinion, trifled with a great national question. All the way along there had been ample evidence of technical and scientific difficulties which rendered it impossible for the present statutes to be effectual. A magistrate in a case was, perhaps, called upon to decide a technical question—it might be as to the percentage of borax that should be permitted in butter. One of their friends might come and declare that 0·5 was a perfectly safe percentage of borax to be introduced, while some more energetic gentleman might declare that 0·7 per cent. was a safe quantity, while the trembling criminal in the dock did not know what a borate was, and the gentleman on the bench probably did not know either, yet he gave a verdict, and fined the man forty shillings. All this had been impressed upon the Select Committee in the valuable evidence that they had been permitted to hear during three sessions, and the conviction grew in their minds that, as such questions were of a scientific and technical nature, it necessarily followed that they must have a scientific Board of Reference to whom these questions should be referred (hear, hear). The Select Committee differed very much among themselves upon certain other details: some of their agricultural friends, for instance, were, figuratively speaking, for hanging all margarine manufacturers, or preventing them from coloring their productions; but they all agreed with perfect unanimity that to deal with these scientific questions it was absolutely necessary that there should be constituted this Board or Court of Reference (hear, hear). But Mr. Chaplin in his Bill altogether ignored this strong and unanimous recommendation of the Select Committee, which was supported by the opinion of all the public bodies that came before them. The Corporations of Glasgow and other important towns sent competent gentlemen to give evidence, who supported the requirement of this Court of Reference. The merchants who came before them also supported it, and the retailer for his own protection demanded it. Even the agriculturists agreed, while analysts of course assented; and, above all, the Government officials themselves agreed. It was, however, ignored altogether, and he could not satisfy his mind as to the cause of its being so ignored, unless it arose from the fact that the Government had altogether a misconception of

the grounds of this great question. He rather imagined that they thought that the pressure which was being brought to bear upon them in Parliament was instigated by the agriculturists in the House of Commons, and he expected that each party was rather suspicious of its agricultural adherents. The agriculturists had certainly made some extreme proposals, and he could not help thinking that the Government might have thought it was the agriculturists who were pushing the movement forward. He said that advisedly, because Mr. Balfour, replying to a question which he (Mr. Kearley) had recently put to him in the House of Commons, had said that it was a very controversial question, and one which raised serious differences between town and country; and he rather imagined that that gentleman also shared the belief that the country people were trying to "run a racket" of their own to the detriment of the towns. Now he (Mr. Kearley) spoke as a townsman, and his views arose from his knowledge of the iniquity of the adulteration that went on unchecked in every town. It was not merely in towns, but in the poorest parts of towns, that adulteration was carried on to the greatest extent. London at that very moment was overwhelmingly full of adulteration, and it was the poor of the Metropolis and the other great centres of England who were the greatest sufferers. He desired himself, if he could, to dissipate the idea that this was a purely agricultural question. It was one which affected the towns more vitally than it did the agricultural districts of the country. So bad had adulteration now become, and so ineffectual had been the operations of local bodies to check it, that it had become necessary for those who were deeply interested in the subject to establish, as it were, their own police. There was in London what was known as the Butter Association, who were running to earth week by week some of these fraudulent gangs. In Lancashire the Agricultural Association had appointed its own inspectors, and was doing good work. According to their report they had taken last year 277 samples, of which no less a number than 135 were adulterated. That was a very serious amount of evidence as to the adulteration that existed. He was sure, from very intimate knowledge of the effects of such unchecked adulteration, that its continuance would ultimately sap the commercial honesty of the country. It was becoming a species of competition. Mr. John Bright, some forty years ago, had described adulteration as a form of competition, which might have been perfectly true in those days; but he (Mr. Kearley) would describe it as a form of robbery, and of the most gross and flagrant character (hear, hear); and the man who was subjected to such competition—if it were called competition, was perfectly entitled to claim the protection of the law. A man who put 30 per cent. of water into milk was a far greater criminal than any poor and starving man who committed a petty theft, although the latter would no doubt receive the heavier punishment. Those who practised adulteration battened and fattened on it, simply because the penalties that were inflicted upon them were quite inadequate to the amount of crime they committed (applause). He would not go further into the details of the abortive Bill that had been introduced by Mr. Chaplin, but he did feel it a pleasure to have the opportunity of supporting before Parliament the cause advocated by the Society of Public Analysts. He had had the honour on two occasions to move an amendment to the Address at the opening of Parliament, and he would look forward to the Queen's Speech with just the same

amount of interest on this occasion as he had done in preceding years. He hoped that there would be found a direct reference in that Speech to the determination of the Government to introduce a Bill, and if there was not, he should feel it his duty to again move an amendment to the Address, and it would be his endeavour and intention, all through the session upon which they were about to enter, to induce the Government, in the interests of Public Analysts, in the interests of consumers, and in the interests of distributors, to bring in a Bill that would be satisfactory to all those who were concerned, and he hoped that if he had the honour to be with the Society again next year, he would be in a position to congratulate them on the success of their endeavours (applause).

Professor TILDEN said that he had accepted with great pleasure the invitation to propose the toast of "The Institute of Chemistry," because, as he thought was well known, he had been very much interested in watching the progress of the Institute during many years. The Institute of Chemistry was established at a time when there did not exist in England any Society or Institute which was prepared to test the qualifications of chemists, and he used this term "chemist" advisedly, notwithstanding that he had had the advantage of sitting during the evening next to his friend, the President of the Pharmaceutical Society. It was well known, of course, that the Pharmaceutical Society, always forward in the field, had some forty years ago got a Parliamentary stamp put upon the type of chemists which it represented, and claimed patent rights in respect of this title. However, he thought he might venture to claim that he was on very good terms with his friend, whom, he had no doubt, the company would find desirous of being on good terms with them. The Chemical Society, which of course was an old Society, having long ago celebrated its jubilee, was approached more than twenty years ago on this question, and it was asked whether a department of the Society could be established, the object of which should be to inquire into the professional qualifications of young chemists, and to put upon them a kind of stamp which would be intelligible to the public. The Chemical Society did not at that time see its way to doing anything of this kind, and so, in 1877, the Institute of Chemistry was established. It went on for a few years, until in 1885 it received a Royal Charter, which for some time, as far as he could remember, did not seem to quite agree with it. A few years later it fell to his lot to offer certain advice, which he thought did it some good, although occasionally the advice that he ventured to offer had been received with rather a wry face. He said that not because he wanted to claim any credit in the matter, but because he wanted to suggest to the Institute that it must take another step or two in order to reach that position which all its fellows and all its well-wishers desired it to occupy. There had been a great deal of talk about its occupying, in regard to the profession of chemistry, a position corresponding to that which was occupied by the College of Physicians towards the profession of medicine, and various other parallels and comparisons had been drawn with it. The Institution of Civil Engineers had been quoted as an instance of the sort of body which it ought to imitate. He ventured to say that the Institute of Chemistry was successfully imitating this greater and older Institution in every respect but one. It was carrying on its work in a thoroughly efficient and satisfactory manner. It was doing that which he considered to be the main object of its

existence, namely, testing the professional qualifications of the younger chemists by a proper system of examination following upon a course of systematic study, and putting upon them a certain stamp; and it gave them a kind of position in the professional world. He did not know that the College of Physicians or the Institution of Civil Engineers did any more than this; but the difference was that the older bodies had command of very considerable monetary resources. He believed that the Institute of Chemistry only required to be in a stronger financial position to become one of the most influential bodies which existed in the Metropolis, or in this country, and, though he really did not know how it was to be done, he suggested to the President and Council, now that they had set their house in order, and had a roof of their own over their heads, and were carrying on work that was of eminent practical value and eminently serviceable to the community, that they ought to proceed somehow or other to get money. Then, he thought, the Institute would be in a really important position, and capable of exercising that kind of influence upon public opinion which at present it was not in a position to exercise. This was the advice that he now ventured to offer to the Institute; how it was to be carried out he left it to the President and Council to take into early consideration. He would not like to conclude without coupling with this toast the name of the present President of the Institute of Chemistry. The present President lived in happy times. The Institute was no longer divided by those sharp contentions, no longer disturbed by those fierce discussions which took place a few years ago, when he (Professor Tilden) was on the Council. The present President had a united Council to work with, and a definite and fixed organization of the Institute and its work to preside over, and he might say that Dr. Stevenson had shown that he possessed the qualities of the best President that the Institute could have. He certainly was a man representing the sort of professional position that the Institute desired; he was a man of peace, and at the same time a man of action, and he (Professor Tilden) was sure that under Dr. Stevenson's guidance the Institute would go on and prosper more and more year after year.

Dr. Stevenson in returning thanks, in the name of the Institute of Chemistry, for the kindness with which this toast had been proposed and received, said that Professor Tilden had omitted one very important fact, namely, that he (Professor Tilden) was himself one of the former Presidents of the Institute who fought those battles of which he had given such a graphic description. He (Dr. Stevenson) felt that as President of the Institute of Chemistry he had indeed fallen upon very happy times. He was present in somewhat of a double character, for he was present as one of the oldest members, and as a former President, of this Society, as well as in the capacity of President of the Institute of Chemistry; and he felt it a great compliment that as soon as he ceased to be President of the Society of Public Analysts he became the President of the Institute of Chemistry. The times, he hoped, were past when there was any feeling of uncordiality between that Society and the one which he officially represented. He ventured to think that the Institute was conferring a great benefit upon the Society of Public Analysts. It had already instituted examinations which, for completeness in testing professional and chemical knowledge, surpassed those of any other society. Its membership was now without question accepted by the Local

Government Board as a complete qualification in everything required of a public analyst except therapeutics, and with regard to therapeutical knowledge he hoped that the gap would soon be bridged over, and that the Institute would formulate a complete qualification for Public Analysts, as far as the Local Government Board was concerned.

Mr. BERTRAM BLOUNT, in proposing the toast of "Kindred Societies," said that among the societies coming under this head the Chemical Society might properly claim the first place. Analysts, unless they received an education in the science of chemistry as distinct from its profession, would have a remarkably poor chance of success, and if they were unable to produce evidence of a knowledge of the science as such, they would not be proper representatives of a great and growing and most honourable profession. He was therefore sorry to have to refer to the absence of the President of the Chemical Society, Professor Dewar, consequent upon lecture engagements in Cambridge; though they welcomed three most eminent members of the Chemical Society, namely, Professor Armstrong, Professor Dunstan, and Mr. Groves. He coupled the toast with the name of the President of the Society of Chemical Industry, Professor Clowes.

Professor CLOWES, in responding, said that in addition to the society which he had the honour to represent, he had to speak on behalf of several other societies, and he had been greatly assisted by the kindness of Mr. Blount in enumerating them. He considered it remarkable that in spite of the increased number of societies, their membership showed no signs of decreasing. He was not aware that the Chemical Society had suffered in any way from the existence of the Society of Chemical Industry, and he was quite sure that the latter society had not suffered in any way from the existence of other societies, the fact being that its membership was increasing faster than ever. Every one of these societies was really required, and each had been constituted to meet an acknowledged need. He returned hearty thanks for the toast on behalf of all the societies that had been referred to, and expressed the hope that they would all continue to work in a friendly way with the Society of Public Analysts.

Mr. WALTER HILLS (President of the Pharmaceutical Society), in proposing the toast of "The Society of Public Analysts," said that probably most of those present were members of the Society of Public Analysts, and would naturally feel a great interest in its prosperity, and he was sure that that prosperity would continue to increase as long as the Society's object continued to be the benefit of the public. His opinion was that no society could flourish unless it existed primarily for the advantage of the public, and he believed that the Society of Public Analysts was doing good work in the interests of the public. It therefore gave him very great pleasure to propose prosperity to it. Mr. Kearley had referred to the Bill which had been recently introduced in the House of Commons by Mr. Chaplin. He (Mr. Hills) had great sympathy with all that Mr. Kearley had said, and would only say in addition, with regard to the calling which he had the honour to represent, that he was sure it would be agreed that, in the Board of Reference which had been recommended by the Select Committee, there should be someone who was representative of the great pharmaceutical interest, for the examination of drugs was attended by more difficulties, perhaps, than the examination of ordinary

articles of food. The subject of milk had often been discussed rather as typical of those matters which came constantly under the purview of the Public Analyst; but the difficulties arising in connection with milk were intensified in the case of drugs. The subject of milk, moreover, was free from the special difficulties arising from the use of synonyms, which were a great bugbear in connection with drugs. Another difficulty was that many drugs were collected by totally unqualified persons, who did not know the right time to collect these various vegetable products, and who sent to this country products possessing widely different pharmaceutical and therapeutical values. With regard to the Society of Public Analysts itself, it had been in existence for some four-and-twenty years, and had been constantly progressing. It was formed for the protection of the public, it had the interests of the public at heart, and it was trying to furnish for the service of the public a number of persons possessing certain special qualifications. It was, moreover, the proprietor of the ANALYST, which was a most valuable periodical. With the proposition of prosperity to the Society of Public Analysts, he had the pleasure of coupling the name of the Society's President.

The PRESIDENT, in acknowledging the toast on behalf of the Society, referred to the self-sacrificing and indefatigable labour of Dr. Sykes in editing the ANALYST, with results which he thought were satisfactory to every reader of that journal, and the readers of the ANALYST were not merely the members of the Society. The paper went abroad all over the world, and was taking up a very strong position wherever the English language was read, and wherever analytical chemistry formed a subject of interest. He hoped that when the time came for him (the President) to finally resign his official connection with the Society, he would be able to congratulate it upon the fact that the great question of the amendment of the Sale of Food and Drugs Act, which had been exercising their attention for so many years, had been finally settled.

Mr. CASSAL proposed the toast "The Guests."

Professor ARMSTRONG, in replying, said that the Society was doing a work on behalf of the public which was of extreme importance. It was not necessary for him to say more than this in returning the guests' most hearty thanks for the toast.

The health of the Honorary Treasurer, Mr. E. W. Voelcker, and of the Honorary Secretaries, Mr. Edward Bevan and Mr. Charles E. Cassal, having been proposed and acknowledged, the proceedings terminated.

ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

FOODS AND DRUGS ANALYSIS.

The Determination of Fat and Water in Cheese. A. Devarda. (*Zeit. anal. Chem.*, 1897, xxxvi., 751-766.)—The author recommends the following as a simple method of obtaining the fat without material alteration and without free fatty acids or lactic acid. From 50 to 100 grammes of the cheese cut in small pieces (or triturated with a little water in a mortar) are mixed with 50 to 80 c.c. of water, and 100 to 150 c.c. of ether in a Wolfbauer shaking flask (a stoppered flask with a small constriction in the middle). Two or three drops of phenolphthalein solution are added, and the whole well shaken with dilute potash until the aqueous layer becomes perceptibly pink. The ethereal layer is then drawn off, the ether distilled, and the fat dried at 100° C., filtered if necessary, and examined in the usual way. A table giving the constants of the fat thus obtained from fifty-one kinds of cheese (including margarine cheese) is given in the paper.

For the determination of the water about 10 grammes of the finely-divided cheese are dried in vacuo over sulphuric acid at the ordinary temperature for 24 to 36 hours, and then for 2 to 6 hours at 100° C., until the weight becomes constant. In this way the bulk of the water is removed at the ordinary temperature, and whilst the method is as speedy, there is no material loss of organic matter (volatile glycerides and acids) such as occurs with long-continued drying at 100° C. Complete drying in vacuo is too tedious, and often impracticable. The following examples show the accuracy of this process:

Cheese.	Loss of Water per cent.			Water per cent.	Per cent. Water.	
	24 hours in vacuo.	A second 24 hours in vacuo.	3 to 6 hours at 100° C.		Dried entirely at 100° C.	Dried entirely in vacuo.
Rodamur ...	46·24	2·24	3·11	51·59	51·92	51·50
Limburg ...	37·79	1·12	0·09	39·00	39·38	38·98
Gervais ...	47·89	—	1·36	49·25	49·36	49·10
Limburg (air-dried)	11·10	—	2·17	13·27	13·46	

C. A. M.

The Composition of some Commercial Powdered Extracts of Liquorice. C. O. Kinsey. (*Amer. Journ. Pharm.*, 1898, lxx., 23-25.)—The method most in use for the determination of glycyrrhizin, on the amount of which the value of extract of liquorice extract depends, consists in exhausting the extract either with boiling water, cold water made alkaline with ammonia, or with a cold mixture of water and alcohol made alkaline with ammonia, and precipitating the glycyrrhizin in the filtrate with dilute sulphuric acid. But the author, finding the directions given by

other chemists vague, and often experiencing considerable difficulty in filtering the insoluble residue, made experiments, and found that the most satisfactory results could be obtained by using as a solvent a mixture consisting of official alcohol 240 c.c., official ammonia water 40 c.c. (*Amer. Pharmacopœia*), and water to make up a litre.

One gramme of the extract was treated with 25 c.c. of the solvent, stirred at intervals for an hour, and left for twelve hours. The supernatant liquid was decanted on to counterpoised filters, the residue washed with 5 c.c. of the solvent, then transferred to the filters, washed till the washings were colorless, and dried in the air-bath at 100° to 110° C.

The filtrate was acidified with dilute sulphuric acid, and the precipitate of glycyrrhizin collected on counterpoised filters, washed until free from sulphuric acid with water containing a little acetic acid, dried in the air-bath at 105° C., and weighed.

The following table gives the percentage results obtained with samples of different commercial brands:

Brand.	Moisture.	Ash.	Insoluble Matter.	Glycyrrhizin.
Spanish	6·52	3·70	36·52	6·40
Greek	6·26	8·18	22·06	14·39
Spanish	5·00	5·51	25·54	10·75
American	5·62	6·79	12·27	7·63
Spanish	7·08	6·52	29·20	5·28
Spanish	6·96	6·56	20·35	10·41
Greek	6·71	7·82	9·65	18·59
—	7·96	5·77	15·21	8·90
—	8·25	5·54	7·40	27·78
—	8·46	4·67	19·41	9·50
—	9·19	6·76	11·12	8·94
—	5·78	7·49	5·95	11·63

(*Cf.* ANALYST, xxii., 192, 219.)

C. A. M.

A Reaction for Chloroform, Bromoform, and Chloral. A. Desgrez. (*Ac. d. Sc.*, 1897, 780; through *Jour. Pharm. Chim.*, 1898, vii., 35, 36.)—Chloroform is decomposed by aqueous potash, giving as the principal products carbon monoxide and water in accordance with the equations:—

$CHCl_3 + 2KOH = 2KCl + H_2O + CO + HCl$ and $CHCl_3 + KOH = KCl + 2HCl + CO$.

The carbon monoxide may be recognised by its blue flame, by its absorption in a solution of cuprous chloride in hydrochloric acid, by Berthelot's test (ammoniacal silver nitrate), and by the result of combustion with oxygen in the eudiometer.

The reaction is accelerated by a gentle heat, and by the presence of light. Bromoform is decomposed in a similar manner, though more slowly, but iodoform, being insoluble in water, is not affected, as is also the case with carbon tetrachloride and methylene chloride. Chloral gives the same reaction as chloroform, but more

rapidly on account of the heat generated by its primary decomposition into chloroform and formate accelerating the action of the potash. Alkaline carbonates and bicarbonates do not cause an analogous decomposition, and ammonia is also without action in the cold. C. A. M.

ORGANIC ANALYSIS.

On Douro Olive Oil. A. J. F. Silva. (*Bull. Soc. Chem.*, 1898, xix., 88-89.)—Milliau was the first to point out that certain kinds of olive-oil contained a coloring matter derived from the aqueous part of the pulp of the fruit which gave a rose coloration with Baudouin's reagent. He also showed that by applying the test to the dried fatty acids instead of to the oil itself, the effect of this might be obviated. Domergue established the same fact in the case of olive-oil from Tunis and Algiers, whilst Villavecchia and Fabris found that some varieties of Italian oil behaved in the same way. Similarly the author finds that the best varieties of Douro oil give a violet coloration with sugar and hydrochloric acid, but that their dried fatty acids give a negative result. The samples examined were perfectly normal in appearance and taste, and as regards their physical and chemical constants.

As Toche's reagent gave no coloration with these oils, the author prefers it to Baudouin's for the detection of sesame-oil in olive-oil. Fifteen grammes of the oil are shaken with an equal quantity of a solution of 2 grammes of pyrogallol in 30 grammes of hydrochloric acid, and after being left for some time, the oily layer is decanted, and the hydrochloric acid warmed for five minutes. The appearance of a reddish-purple colour denotes the presence of sesame-oil. C. A. M.

Testing Oil Paints. H. Loesner. (*Chem. Rev. Fettu. Harz-Ind.*, iv. [24], 329-330.)—In order to determine the permeability of a paint, and consequently gauge its value as a protective coating for iron, the author proposes to employ a steam test. A thin, uniform layer of the paint is applied to the surface of a polished strip of sheet-iron (2 by 4 inches), followed by a second coating when the first is dry. The strip, when dry, is then laid, with the painted side downwards, over the opening in a water-bath, and supported by strips of wood (to prevent mechanical injury) so that the surface of the paint is two inches above the constant level of the water. After exposure to the steam of the bath for fifteen hours, the strip is dried at a maximum temperature of 100° C., and after loosening and removing the paint with aniline, its surface is examined for rust.

Resistance to acids may be tested by exposing the painted strips to the fumes of 33 per cent. hydrochloric acid at 20° C., the distance between the surface of the paint and the level of the acid being the same as in the steam test. The incrustation formed on the paint must be removed by washing at intervals, and at the end of twelve hours the paint is removed as before and the metal examined for corrosion.

The steam test is also useful for examining fats, vaseline, lacquers, etc., intended

for use as rust preventors. Any of these substances that will stand the test for four to five hours may be regarded as satisfactory, but for oil paints twelve hours is the minimum exposure permissible. C. S.

Characteristics of some scarce Resins. K. Dieterich. (*Chem. Rev. Fett u. Harz-Ind.*, vol. iv. [24], 327-329.)

Crude Chicle Gum, obtained from *Achras Sapota*, is particularly rare. It contains 70 per cent. of resin, 10 per cent. of gum, 9 per cent. of calcium oxalate, 5 per cent. of sugar and inorganic salts. The pure resin can be extracted by carbon disulphide; it forms a plastic mass, which resembles guttapercha, and can be vulcanized.

Guaiacum in lacrymis, another scarce resin, gives an acid number, 72·8-75·6, by the author's method, whilst that of the ordinary guaiacum is 89·6-92·5.

Socotra Dragon's Blood. The pure red resin from this variety is a mixture of several resins, and differs from Palm Dragon's blood in lacking the draco-alban, characteristic of the latter. Apart from this and their varying solubilities, little difference exists between their other characteristics.

Bisabol Myrrh differs from the ordinary Herabol myrrh of commerce. To prevent errors—arising from the examination of the alcoholic extract instead of the natural drug—the author proposes the following method of determining:

1. *The Acid Number.* One gramme of a very finely powdered average sample of the myrrh is heated along with 30 c.c. of water for a quarter of an hour under a reflux condenser, and then for a similar period with 50 c.c. of strong alcohol. After cooling, the liquid is titrated with $\frac{N}{2}$ alcoholic potash, using phenolphthalein as indicator.

2. *Saponification value.* One gramme of an average sample is infused in 30 c.c. of water, and after standing for half an hour, mixed with 33 c.c. of $\frac{N}{2}$ alcoholic potash, boiled for half an hour under a reflux condenser, then cooled, diluted with alcohol, and titrated back.

3. *Ester number* by difference as usual.

The following values were obtained for the two varieties:

	Acid Number.	Ester Number.	Saponification Value.	Solubility in Alcohol.
Bisabol Myrrh	20·06	125·54	145·6	50 per cent.
Herabol Myrrh	25·48	204·12	229·6	20 per cent.

C. S.

The Preparation of Absolute Alcohol by means of Calcium Carbide. P. Yvon. (*Jour. Pharm. Chim.*, 1898, vii., 100-102.)—Calcium carbide furnishes a ready means of determining whether alcohol is anhydrous or not. On adding a pinch of the powder to several c.c. of absolute alcohol, no bubbles of gas are liberated and the liquid remains transparent, whilst if only a trace of water is present, bubbles of acetylene are liberated, and the liquid becomes milky from the formation of calcium hydrate.

In order to prepare absolute alcohol from 95 or even 90 per cent. alcohol, the latter

is mixed with about one-fourth of its weight of powdered calcium carbide, shaken at intervals during two or three hours, and then left for twelve hours, when all liberation of acetylene should have ceased. It is then cautiously distilled, the first portion, which contains acetylene, being rejected. The alcohol obtained is usually anhydrous, but it is advisable to redistil after the addition of a little copper sulphate to remove acetylene. C. A. M.

Reducing Action of Mineral Acids on Fehling's Solution. M. Z. Jovitschitsch. (*Berichte*, 1897, 2,431.)—The author finds that the addition of small quantities of the mineral acids to Fehling's solution by lessening its alkalinity effects its reduction, even when the liquid is still alkaline to litmus paper, just as effectually as such reducing bodies as glucose, phenylhydrazin, etc. Often the action commences before the liquid is warmed, as in the following case: If to 1 or 2 c.c. of Fehling's solution a few drops of a dilute mineral acid are added in quantity insufficient to discharge the blue colour, and then a few drops in the original Fehling, a turbidity indicating reduction immediately appears, which may be completed by gently heating the liquid. Tartaric acid, employed in a similar manner, also causes reduction. The cause of this is supposed to be the decomposing action of the mineral acid on the tartaric acid of the Fehling's solution; and the reduction which occurs when this solution is kept for some time in well-closed bottles is attributed to spontaneous decomposition of the tartaric acid. W. J. S.

The Chemical Examination of Saliva. E. Gerard. (*Jour. Pharm. Chim.*, 1898, vii., 12-15.)—This paper deals more especially with a pathological case in which an excess of saliva was secreted by an epileptic patient, but also contains remarks on the composition and analysis of normal saliva. The analytical results obtained by the author were: Density at 15° C., 1·003; total solids, 7·85 per cent.; ash, 4·80 per cent.; substances precipitable by alcohol, 2·30; mucin and albumin, traces.

The proportion of salts here is higher than in normal saliva, and bears out Heidenhain's law that the more rapid the secretion, the greater the percentage of mineral matter.

It has been shown by several chemists (*e.g.*, U. Gautrand, *Thèse de Lyon*, 1895) that the amylolytic power of saliva shows a considerable variation under the influence of different diseases. For this determination Jawein's method (*Wien. Med. Press*, xxxiii., 626) was adopted. To 100 c.c. of a 4 per cent. solution of starch 4 c.c. of the saliva were added, and the mixture kept at 39° to 40° for fifteen minutes. It was then diluted to 200 c.c., and the percentage of maltose determined. This varied on different days from 0·603 to 0·639 gramme, whereas normal saliva yields from 0·368 to 0·555 gramme.

The temperature at which the ptyalin became inactive was determined on the ptyalin itself, obtained by precipitation with alcohol and dissolved in water, following the directions of E. Bourquelot (*Ferments Solubles*, 1896, 136). In the author's case the ptyalin was still active at 57° C., became enfeebled at 58° to 59° C.,

and inactive at 60° to 61° C. In this respect it behaved like the ptyalin of normal saliva.

This saliva contained an oxidizing ferment, which has also been found in the normal secretion by P. Carnot (*C. R. Soc. Biol.*, May 29, 1896). C. A. M.

The Influence of Certain Drugs on the Analysis of Urine. B. Bardach. (*Zeit. anal. Chem.*, 1897, xxxvi., 776-784.)—I. *The Influence of Nitrates on the Determination of Nitrogen by Kjeldahl's Method.*—In order to obviate the loss which occurs when the nitrogen in urine containing nitrates is determined either by Kjeldahl's original method, or by the Jodlbauer-Foster modification, or with the addition of phenol to the sulphuric acid, the author has devised the following process, in which the nitrates are first reduced in alkaline solution: Ten c.c. of the urine are placed in a 500 c.c. Kjeldahl flask, together with about 0·3 gramme of aluminium strips (previously heated), 20 c.c. of water and 5 c.c. of soda-lye (specific gravity 1·34) added, and the flask rapidly connected with an absorption flask containing a known quantity of standard sulphuric acid. The flask is left for about forty-five minutes, being occasionally shaken, and is then heated on a wire gauze over a small flame for about five minutes, until the development of gas has practically ceased. The flask is then cooled by drawing air through it, 30 c.c. of concentrated sulphuric acid added (and subsequently some potassium permanganate), the liquid heated until colorless, and the ammonia distilled over in the usual way.

The following are some of the results thus obtained, calculated on 100 c.c. of the urine:

Nitrogen of the Urine. Grammes.	Added Nitrogen in the form of Nitre. Gramme.	Total Nitrogen Calculated. Grammes.	Total Nitrogen Found. Grammes.
0·884	0·348	1·23	1·25
0·788	0·069	0·857	0·849
1·000	0·173	1·173	1·169

II. *On the Determination of Urea by Liebig's Method.*—Salkowski (*Pflüg. Arch. f. Phys.*, vi., 244) pointed out that potassium iodide had an influence on the determination of urea, since, owing to the formation of mercuric silver iodide the end-point of the reaction occurs too soon, and the amount of urea found is too low. This error, however, is considerably modified by the addition of sodium chloride, and owing to the presence of that salt in normal urine is comparatively trifling.

The author finds that ammonium chloride has a similar disturbing influence, but in the opposite direction, delaying the end-point of the reaction, and causing the amount of urea to appear too high. This error is considerable with little more than 0·3 per cent. of ammonium chloride, and with larger quantities the results are entirely worthless.

III. *On the Determination of Uric Acid.*—Experiments are described which prove that the usual doses of lysidin and piperazine have no appreciable effect on the results of the determination of uric acid by Salkowski's method (*Prak. d. phys. u. path. Chem.*, 239). Similarly in the case of potassium iodide the author obtained correct results, although great care was necessary to avoid loss. C. A. M.

INORGANIC ANALYSIS.

Solubility of Copper Sulphide in Sodium Sulphide. A. Hollard. (*Ann. de Chim. Analyt.*, ii. [13], 242.)—By digesting out of contact with the air pure precipitated copper sulphide for a sufficient time with concentrated sodium sulphide (specific gravity 1·225), prepared according to the directions given by Classen, and saturated with hydrogen sulphide, it was found, after filtering the liquid with due precautions to prevent oxidation and throwing down the dissolved copper by electrolysis, that the maximum amount of copper in the condition of sulphide that can be dissolved by 100 c.c. of sodium sulphide is 0·0032 gramme. C. S.

The Separation of Thorium from Cerium. E. Hintz and H. Weber. (*Zeit. anal. Chem.*, 1897, xxxvi., 676-685.)—After experimenting with different analytical methods, the authors have arrived at the conclusion that the separation of thorium from cerium by means of sodium thiosulphate is not absolute, a small amount of thorium remaining in the solution and of cerium in the precipitate, but the errors resulting from this are only trifling, and to a large extent compensate one another.

The separation by means of ammonium oxalate is also incomplete in a single precipitation, and, although better results can be obtained by repeated precipitation, yet the amount of cerium formed is still slightly too high in spite of the fact that traces of cerium can be detected in the filtrate. Very small quantities of thorium oxalate, however, can be completely dissolved by one treatment.

Glaser's method (ANALYST, xxi., 274), based on the solubility of thorium oxalate in a solution of ammonium oxalate containing ammonium acetate, has, according to the authors' experiments, the same drawback, and they recommend the thiosulphate method as being the most satisfactory for the analysis of incandescent mantles and thorium nitrate. The most accurate results are obtained by adding ammonia to the filtrate from the thiosulphate precipitation, dissolving the precipitate in acid, expelling the latter by evaporation, adding oxalic acid, and extracting the small amount of thorium oxalate with ammonium oxalate. C. A. M.

On the Determination of Insoluble Phosphorus in Iron Ores. C. T. Mixer and H. W. Dubois. (*Jour. Amer. Chem. Soc.*, 1897, xix., 614-619.)—The following simple process is based on the fact that ignition of the residue insoluble in hydrochloric acid converts the insoluble phosphorus into a soluble form: About 1½ gramme of the ore is treated with 25 c.c. of hydrochloric acid (specific gravity 1·10), and when as much as possible has dissolved, the excess of acid is expelled by evaporation, and the liquid diluted and filtered. The residue and filter paper are ignited in a platinum crucible at a red heat for a couple of minutes, the mass being stirred with a platinum rod. It is then placed in a beaker with a little water and a few drops of hydrochloric or nitric acid, the liquid gently boiled for about five minutes, filtered into the flask containing the first filtrate (or into another flask if this portion is to be determined separately), neutralized with ammonia, and the phosphorus precipitated as ammonium phosphomolybdate and titrated.

The results obtained in this way are in close agreement with those given by the older method of dissolving the ore and fusing the residue with sodium carbonate. The authors' objections to the latter are, that a longer time and larger crucibles are required for the fusions, and that the introduction of a sodium salt is often unfavourable to the obtaining of a pure precipitate of ammonium phosphomolybdate.

C. A. M.

Determination of Citrate-Soluble Phosphoric Acid in Thomas Phosphate Meal. O. Böttcher. (*Chem. Zeit.*, xxi., 993.)—In a recent paper on the valuation of phosphates according to their citrate-solubility (*Chem. Zeit.*, xxi., 905; ANALYST, xxii., 334), P. Wagner shows that the phosphoric acid in a citrate-extract of Thomas phosphate can be precipitated free from silica direct by magnesia, without the intervention of the molybdenum process, provided that the alkaline citrate and magnesia mixture are added while the extract is fresh. The author claims priority for this observation, as in a paper sent to the *Chem. Zeit.* about a month before Wagner's communication he stated that it was necessary that the citrate solution and magnesia mixture should be added as soon as possible after the extract was ready—at the latest within the same day.

Wagner further states in his paper above referred to that it makes no difference whether the ammonium magnesium phosphate be filtered off immediately after stirring, or after standing for two hours. The author, remarking upon this, observes that he has already, in an earlier paper, called attention to three cases in which silica was deposited in citrate-extracts of Thomas phosphate meal on standing for two hours after precipitation, thus seriously vitiating the results. Though such cases may be rare, it is nevertheless necessary to take into consideration the possibility of their occurring, and therefore the filtration should be carried out promptly. It is also important that the shaking should be commenced immediately after the addition of the citrate solution and magnesia mixture. H. H. B. S.

Wagner's Method for the Determination of Citrate-Soluble Phosphoric Acid in Thomas Phosphate Meal. B. Sjollema. (*Chem. Zeit.*, xxi., 999.)—The author recommends the following procedure: To 50 c.c. of citrate-extract, prepared according to Wagner's directions, are added 50 c.c. of an alkaline citrate solution (containing in 5 litres, 500 grammes citric acid, and 4 litres ammonia of 0·96 specific gravity) and 15 c.c. of magnesia mixture (containing in 2 litres, 110 grammes magnesium chloride, 140 grammes ammonium chloride, and 700 c.c. ammonia of 0·96 specific gravity). The solution is shaken for ten minutes, allowed to stand for two hours, or until the following day, and filtered off. In very few cases does any silica separate. When, however, this difficulty is encountered (which is evidenced by the appearance of the precipitate and the slowness of the filtration), the determination is recommenced, and the solution shaken for half an hour and then at once filtered off. In many cases the author tests the pyrophosphate for silica after weighing by dissolving in hydrochloric acid. H. H. B. S.

Gravimetric Method of Estimating Phosphoric Acid as Ammonium Phosphomolybdate. T. S. Gladding. (*The American Fertilizer*, 1898, i.)—To the solution (25 c.c. to 50 c.c., corresponding to 0·25 gramme of substance) is added 25 c.c. of ammonia of 0·9 sp. gr., and sufficient nitric acid of 1·42 sp. gr. to neutralize the ammonia and produce an acid reaction. The beaker containing the solution is then placed in a water-bath, heated to a temperature of 50° C., and about 10 c.c. excess of ordinary 5 per cent. molybdate solution added from a burette at the rate of about three drops per second, with constant stirring. The beaker is allowed to remain for a further ten minutes in the bath, and the solution is then filtered off through a weighed filter. The filtrate (without the washings) should remain clear, or only show a slight opalescence, on the addition of 5 c.c. of molybdate solution, and further heating for ten minutes in the bath. The precipitate is washed three times by decantation, and three times on the filter with dilute nitric acid (100 : 1), followed by one wash with water. The filter is then drained on blotting-paper and dried at 105° C. The presence of so large a quantity of ammonium nitrate ensures complete precipitation, and by the low temperature employed the separation of molybdic oxide or iron salt is avoided. For the final drying at 105° C. a bath containing dilute glycerol (1·160 sp. gr.) is used.

Experiments undertaken to determine the exact composition of the yellow precipitate dried at 105° C. gave results corresponding to the formula

$$24MoO_3,P_2O_5,3(NH_4)_2O + 24MoO_3,P_2O_5,2(NH_4)_2O,\ H_2O + 5Aq.$$

H. H. B. S.

On the Determination of Phosphoric Acid as Magnesium Pyrophosphate. H. Lasne. (*Bull. Soc. Chim.*, 1897, xvii., 823-832.)—In this paper the author describes at length his experiments on the accuracy of the determination of phosphoric acid by precipitation as a magnesium salt. He finds that rapid precipitation gives an excess of magnesium in the precipitate owing to the partial formation of trimagnesium phosphate, which only becomes transformed into ammonium magnesium phosphate after contact for sixteen hours with ammonium citrate in sufficient concentration (10 grammes in 150 c.c. of liquid). It is necessary, therefore, in order to obtain absolutely accurate results, to allow the precipitate to stand overnight. But this source of error is too trifling to lead to the rejection of the well-known rapid method (which the author describes as "citro-mechanical") for industrial purposes. The transformation of tri-magnesium phosphate into ammonium magnesium phosphate is very slow in the presence of ammonium chloride alone, and the amount of citrate mentioned above ought always to be added.

The precipitation of magnesium by means of an excess of ammonium phosphate gives at the same time as the ammonium magnesium phosphate a phosphate containing less magnesium, and the greater the excess of phosphate, the less the magnesium obtained in the precipitate. This is shown in the following tables, in which the results were obtained by dissolving a known quantity of strongly calcined magnesia in hydrochloric acid, and precipitating it as phosphate in the presence of ammonium citrate:

	A. Gramme.	B. Gramme.		C. Gramme.
Magnesia taken	0·0948	0·1034	Magnesia taken	0·1034
Corresponding to pyrophosphate	0·2630	0·2868	Corresponding to phosphoric acid	0·1834
Weight of calcined precipitate obtained with			Phosphoric acid in the precipitate obtained with	
5 c.c. amm. phosph. (10%)	0·2687	0·2929	5 c.c. of amm. phosph.	0·1940
10 c.c. ,, ,,	0·2709	0·2981	10 c.c. ,, ,,	0·1971
20 c.c. ,, ,,	0·2779	0·3107	20 c.c. ,, ,,	0·2064

From this the author concludes that the results obtained by this classic method of estimating magnesium are always erroneous. C. A. M.

Separation of Crystallized Silica (Quartz) from the Amorphous Variety. G. Lunge and C. Millberg. (*Monit. Scient.*, 1897, [4], xi., 867; through *Chem. Zeit. Rep.*, 1897, 324. Also *Zeits. angew. Chem.*, 1897, 393 and 425.)—In spite of Michaelis's statement to the contrary, caustic alkalies cannot safely be employed to separate amorphous silica from quartz, as they dissolve notable quantities of the latter. Even warm sodium carbonate acts quickly and appreciably on quartz when it has been reduced to a very fine state of subdivision. But in a powdered material, which does not contain any real dust, the solvent powers of a 5 per cent. solution of sodium carbonate are almost entirely limited to the amorphous silica; and the maximum error likely to occur during such a separation will be a deficit of 0·1 to 0·2 per cent. of the total silica on the side of the quartz.

If silicate minerals are powdered as finely as possible, treated with hydrochloric acid, dried at 110° C., and then digested for fifteen minutes with 5 per cent. sodium carbonate on the water-bath, all the silica set free by the acid will be separated from the quartz and the unattacked silicates, but the amount of the former will probably be 0·2 per cent. too high.

Trass and puzzoliana do not contain any free amorphous silica. They are composed partly of quartz and inactive silicates; while their active ingredients consist of zeolites and, in particular, of a silicate of sodium and aluminium, resembling analcite, which is completely soluble on the water-bath in 30 per cent. caustic potash. F. H. L.

The Use of Sodium Oxalate in Acidimetry. S. P. L. Sorensen. (*Zeit. anal. Chem.*, 1897, xxxvi., 639-643.)—Normal sodium oxalate is readily obtained in a pure state by crystallizing the commercial salt from a feebly alkaline solution, washing the crystals with cold water and recrystallizing them. It contains no water of crystallization, is not hygroscopic, and can be weighed out very exactly after being dried at 125° C. or even 150° C. The weighed quantity is then converted into sodium carbonate by moderate ignition in a covered platinum crucible.

Sodium oxalate can also be used for standardizing permanganate solutions, and has the advantage over oxalic acid of not effloresceing. C. A. M.

THE ANALYST.

APRIL, 1898.

PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS.

THE monthly meeting of the Society was held on Wednesday evening, March 16, in the Chemical Society's Rooms, Burlington House, the President (Dr. Bernard Dyer) occupying the chair.

The minutes of the previous meeting were read and confirmed.

Papers were read on "The Composition of Milk and Milk Products," by H. Droop Richmond; "The Calculation of Added Water in Adulterated Milk," by H. Droop Richmond; "Japanese Wood Oil," by J. H. B. Jenkins; "A Typical North-East Lancashire River," by F. R. O'Shaughnessy; and "The Analysis of Marmalade," by L. K. Boseley; and a new form of condenser was exhibited by Mr. C. H. Cribb, B.Sc.

The following gentlemen were elected members of the Society: Mr. H. H. Dains, F.I.C., Mr. A. D. Hall, M.A., Dr. Otto Rosenheim, and Dr. Philip Schidrowitz; and Messrs. A. P. Davson and C. A. Hackman were elected associates.

NOTE ON THE TESTS FOR DISTINGUISHING BOILED FROM UNBOILED MILK.

BY HENRY LEFFMANN.

(*Read at the Meeting, February* 2, 1898.)

IN the ANALYST for August, 1897, p. 211, is a brief note taken from the *Jour. Pharm. et Chim.*, giving several tests for distinguishing between raw and boiled milk. I have repeated these tests under varying conditions, and also tried other analogous reagents. As noted in the original article, the most striking reaction is with 1 : 4 diamidobenzene. When a freshly prepared dilute solution of this substance is added to raw milk, and then a few drops of hydrogen dioxide solution, a deep blue colour at once appears. I have found that the colour is produced to a marked degree when the milk has been heated to 170° F. (76·5° C.), but after heating to 180° F. (82° C.) the property is lost. It seems possible, therefore, to distinguish between pasteurized and sterilized milk. Whole milk, ordinary skimmed and separator-skimmed milk, exhibit substantially the same colour. In addition to the reagents noted in the original report, I have found that the photographic developer, amidol, can be used,

but eikonogen and various amido-, hydroxy-, and carboxy-derivatives of benzene and naphthalene were found to be inactive.

Sour raw milk still shows the reaction, and even when more advanced decomposition has set in, but in the latter case it is less striking.

Some experiments were made with a view to determining what ingredient in the milk causes the reaction. The temperature—about 175° F.—which destroys the effect is about that at which enzymes lose their activity. Various commercial enzymes, malt-diastase, taka-diastase, peptenzyme, pancreatic extract, papaw ferment, pine-apple ferment, and rennet ferment were used without effect. Solutions of blood- and egg-albumin also gave negative results. A sample of raw milk was treated with excess of magnesium sulphate, and the precipitate filtered off. The filtrate gave a deep blue colour with the 1 : 4 diamidobenzene, and a red tint with amidol, but no effect with the other reagents.

A freshly-opened sample of Eagle condensed milk gave no colour. A piece of cheese rubbed up with water also failed to give the reaction.

A solution of the diamidobenzene, after standing an hour or more, gives a slight blue colour with milk, boiled as well as raw, without the addition of hydrogen dioxide, consequently the solution for the test must be freshly made.

COPPER "PURE FOR ANALYSIS."

By James W. Westmoreland, F.I.C., *Associate of the Royal School of Mines, London.*

(*Read at the Meeting, February* 2, 1898.)

Some time ago circumstances which need not be detailed led me to examine a sample of copper-foil, "pure for analysis," which had been obtained from a firm of wholesale chemists. I found it to contain copper, 99·30 per cent. The metal also contained ·02 per cent. of lead, and some arsenic, but was not further examined.

Later I called on the sellers, and found that their representative was by no means ready to admit the impurity of the metal; he stated that it had been supplied to many analysts, but none except myself had complained of the quality. Assuming this copper to be pure, and using it for standardizing volumetric solutions, it is evident that concordant yet very erroneous results might be obtained. For example, in testing material containing 5 per cent. of copper, the results would be 0·035 per cent. too high, with 70 per cent. material 0·49 per cent. too high, while metal containing over 99·3 per cent. would apparently contain over 100 per cent. copper.

I next obtained some "pure copper-foil" from another dealer after requesting the assistant to be careful and give me the pure material. He pointed out the beautifully-planished surface of the metal as a guarantee of its purity, stating that such care would not be taken with ordinary copper-foil. The metal was well planished, but, as in many other cases, the outward appearance was no guarantee of inward purity. It contained copper 99·10 per cent.

It is just possible that the vendor heard indirectly the result of this test, for another sample purchased later was practically pure.

Another firm stated that their pure copper-foil was not electrolytic copper, but was probably nearly as pure, and this turned out to be the case. From another firm samples were obtained marked "Copper-foil Opt," which contained copper 99·81 per cent., and a sample of "electrolytic copper," prepared from purified sulphate of copper, which I have taken as one of my standard samples.

From another London dealer I obtained some copper marked "Free from Arsenic;" this is a specific statement of quality, but on examination I found the metal to contain:

Copper	... ...	99·23 per cent.
Arsenic	... ...	0·67 ,,

The following samples were obtained from a city in the North of England, marked "Copper, pure for Analyses":

(*a*)	Copper	... ...	99·29 per cent.
(*b*)	,,	... ...	99·64 ,,
(*c*)	,,	... ...	99·87 ,,

Other provincial samples gave

(*d*)	Copper	... ...	99·03 per cent.
(*e*)	,,	... ...	99·85 ,,
(*f*)	,,	... ...	99·52 ,,
(*g*)	,,	... ...	99·65 ,,
(*h*)	,,	... ...	pure.

Another sample, described "as pure as it is made," contained copper 99·38 per cent. Another sample, marked "Free from Arsenic," contained copper 99·60 per cent., but was not further examined. I also obtained samples of metal which is sold as "high conductivity copper," and obtained the following results:

(*a*)	Copper	... ...	99·86 per cent.
(*b*)	,,	... ...	99·83 ,,
(*c*)	,,	... ...	99·84 ,,

I have used the electrolytic and iodide methods for determining the percentage of copper in the various samples. It is evident that although samples which give 100 per cent. by the electrolytic test may possibly not be pure, samples which give lower results, say 99·5 per cent., cannot possibly be pure. The samples stated to be "pure" gave practically 100 per cent. by electrolytic test, and gave results agreeing within the error of experiment when used for standardizing thiosulphate solutions for the iodide test, in conjunction with copper obtained by the electrolysis of a solution of the pure sulphate, and of another sample of extremely pure copper, of which I obtained some quantity. Some of the impure samples were also used in this way (taking the percentage found by the electrolytic test)—and gave results agreeing within the limits of experimental error.

Addendum.

Since my paper was read I have obtained two more samples of "electrolytic copper foil," pure for analysis, from a London firm, which contained 99·00 and 99·09 per cent. of copper respectively.

I have also obtained a sample of "pure copper foil" from a provincial dealer, which gave the following extraordinary results on analysis:

Copper	94·42
Lead	0·39
Zinc	4·91
Iron	0·26
Arsenic	traces
	99·98

I submitted a sample of this metal to a firm of London metal workers, who informed me that it was "pure bright rolled copper." The vendor, in answer to inquiries, stated that the metal was obtained some time ago from a London dealer, and that it was marked "Electrolytic copper foil—pure for analysis—free from arsenic."

DISCUSSION.

Mr. BLOUNT said that copper was a substance pre-eminently easy to obtain in a state of purity almost absolute. Nevertheless, the figures obtained by Mr. Westmoreland showed a condition very far removed from purity. At the same time, although this indictment lay against copper sold as "pure for analysis," he could not help thinking that Mr. Westmoreland must have been a little unfortunate in his samples. Copper containing 99·1 or 99·3 per cent. of actual Cu, and sold as pure, was surely a somewhat unusual material, when it was considered that ordinary locomotive firebox copper contained 99½ per cent.—that was to say, it was not only specified to contain, but actually did contain, that quantity, and very often considerably more. He thought it would be interesting to know what was the balance of the impurities in each case. For instance, in the first sample referred to in the paper, containing 99·3 per cent., Mr. Westmoreland had found some arsenic, but had not said how much, or of what the balance consisted. Beside the arsenic, there was presumably something else. The percentage of arsenic found in the sample sold as "free from arsenic," viz., 0·67, was enormous in a refined tough copper, but even in this case a balance of 0·1 per cent. remained unaccounted for. It seemed curious that so many of these arsenical coppers consisted mainly of copper and arsenic, being tolerably free from other impurities. Ordinary tough-pitch copper, containing 99·5 per cent. of actual Cu, and perhaps 0·2 per cent. of lead, would contain possibly 0·15 per cent. of arsenic. At the other end of the scale there might be 0·4 of arsenic, with 99·5 per cent. of Cu, all the other impurities being included in the remaining 0·1. It was just conceivable that some of the samples examined by Mr. Westmoreland were refined but not toughened coppers, and in such case they might very well have been free from most impurities save oxygen. It was impossible, however, to roll into foil copper containing much oxygen, so it seemed fair to assume that foil was not "refined" as distinct from "tough-pitch." The largest quantity of oxygen that could be contained by copper-foil would be about 0·15 or 0·2 per cent., so that the balance left from this and the actual Cu found by Mr. Westmoreland would probably be lead, arsenic, nickel (a common impurity in commercial copper), and the other odds and ends of things generally found. Touching the question of analysis, it was clearly

evident that to obtain reliable results one must either be prepared to isolate the whole of the copper (and he knew of no process of doing this with any degree of accuracy except electrolysis), or one must have a standard for comparison which was unexceptionable. He gathered that Mr. Westmoreland had followed both modes of procedure, and in that case his figures would be gratefully received. He might mention, however, that in the electrolytic method it was necessary, in order to avoid serious errors, to isolate the copper as nearly as possible from a large quantity, *e.g.*, 10 grammes of the sample, and then to determine the residual copper in the solution.

Mr. WESTMORELAND said in reply that, with the exception of one sample of electrolytic copper prepared from the pure sulphate and the three samples of "high conductivity" copper wire, all the samples were copper-foil. The samples were obtained from practically every dealer in the United Kingdom. His (Mr. Westmoreland's) primary object was to obtain pure copper for standardizing volumetric solutions; consequently he had not troubled in most cases to further examine samples when he found they were impure. The absence of other impurities in arsenical coppers containing, as Mr. Blount suggested, 99·5 per cent. of copper and 0·4 per cent. of arsenic, may be explained on the assumption that they are produced from very arsenical blister copper, and that, consequently, the other impurities present are eliminated before the arsenic is reduced to an allowable percentage. He (Mr. Westmoreland) had many years ago pointed out the necessity of determining the residual copper in the solutions from electrolytic assays.

THE COMPOSITION OF MILK AND MILK PRODUCTS.

By H. DROOP RICHMOND.

(*Read at the Meeting, March* 16, 1898.)

Of the 35,007 samples analysed during 1897 in the Aylesbury Dairy Company's laboratory, 28,762 were samples of milk.

The average composition of 12,907 of these, taken on arrival of the milk from the farms at the depots, is given in Table I.

TABLE I.—AVERAGE COMPOSITION OF MILK DURING 1897.

	Morning Milk.				Evening Milk.				Average.			
	Specific Gravity.	Total Solids.	Fat.	Solids-not-Fat.	Specific Gravity.	Total Solids.	Fat.	Solids-not-Fat.	Specific Gravity.	Total Solids.	Fat.	Solids-not-Fat.
January ...	1·0327	12·75	3·69	9·06	1·0326	13·16	4·07	9·09	1·0327	12·95	3·88	9·07
February ...	1·0326	12·65	3·63	9·02	1·0325	13·04	3·99	9·05	1·0325	12·85	3·82	9·03
March ...	1·0325	12·59	3·60	8·99	1·0324	12·96	3·95	9·01	1·0324	12·78	3·78	9·00
April ...	1·0325	12·54	3·58	8·96	1·0323	12 93	3·93	9·00	1·0324	12·73	3·75	8·98
May ...	1·0328	12·38	3·38	9·00	1·0324	12·92	3·91	9·01	1·0325	12·65	3·65	9·00
June ...	1·0325	12·29	3·37	8·92	1·0319	12·81	3·91	8·90	1·0322	12·55	3·64	8·91
July ...	1·0318	12·32	3·53	8·79	1·0314	12·68	3·92	8·76	1·0316	12·50	3·73	8·77
August ...	1·0315	12·42	3·67	8·75	1·0311	12·81	4·07	8·74	1·0313	12·61	3·87	8·74
September ...	1·0321	12·59	3·68	8·91	1·0316	13·05	4·17	8·88	1·0319	12·82	3·93	8·89
October ...	1·0324	12·59	3·62	8·97	1·0321	13·04	4·06	8·98	1·0323	12·81	3·84	8·97
November ...	1·0324	12·68	3·70	8·98	1·0320	13·20	4·22	8·98	1·0322	12·94	3·96	8·98
December ...	1·0325	12·75	3·74	9·01	1·0324	13·17	4·15	9·02	1·0328	12·96	3·95	9·01
Average ...	1·0324	12·54	3·60	8·94	1·0320	12·98	4·03	8·95	1·0322	12·76	3·82	8·94

It is seen that the composition differs but very slightly from that found in 1896. As usual, the lowest fat was found in June, and the highest during the latter months of the year, and a marked depression of solids-not-fat was noticed in July and August.

The methods used in 1896 have been employed without any modification; in the few cases where the solids-not-fat fell below 8·5 per cent., milk-sugar, proteids, and ash have been determined. The following samples are the only ones showing any abnormality, and were passed as genuine, though not authenticated:

	I.	II.	III.
Specific gravity ...	1·0294	1·0288	1·0355
Total solids ...	12·63	12·34	13·63
Fat	4·35	4·20	3·80
Sugar	4·14	4·13	4·67
Proteids	3·38	3·29	4·35
Ash	0·76	0·71	0·77
Solids-not-fat ...	8·28	8·14	9·83

I have looked through the results of analyses for many years past, and have compiled the following table of the probable number of samples which will be found per 100, having solids-not-fat between the limits named. These figures only refer to the mixed milk of herds:

Percentage of Solids-not-Fat.	Probable Number per 100.
8·4 to 8·5	1·892
8·3 ,, 8·4	0·242
8·2 ,, 8·3	0·027
8·1 ,, 8·2	0·022
8·0 ,, 8·1	0·008
Below 8·0	0·002

It is seen that the proportion of samples appreciably below 8·5 per cent. is excessively small; the bulk of these occur during the months of July and August. I am not in possession of sufficient data to say definitely that the few genuine samples with solids-not-fat below 8·5 per cent. can be distinguished from adulterated milks; but the whole of the samples low in solids-not-fat that I have examined, with whose genuineness I am satisfied (forty-six in all), have had an ash above 0·70 per cent., and nitrogen above 0·50 per cent. On the other hand, all samples to which I have added water have been below those limits, as well as below 8·5 per cent. of solids-not-fat.

I would urge on public analysts the importance of determining the ash and proteids (or nitrogen) on every sample which, from their preliminary results, they consider suspicious.

The preparation of ether for milk analysis has received some attention; the occurrence in commercial ether of an acrid substance, which appears to be obstinately retained by fat, has probably been noticed by all. My first attempts at removing this were by distillation from oil, with a fair amount of success; I have, however, found that by distillation of the ether on a water-bath not exceeding 40° in temperature, from a flask fitted with Glynsky's bulbs, a product is obtained which is eminently satisfactory; the last fraction (B.P. 34·6° to 34·8°) can be used for thermo-regulators

for incubators, as at 37° to 38° it has a vapour tension of about 2½ inches of mercury above atmospheric pressure.

In the analysis of cream and clotted cream amyl alcohol has been substituted for ether to dissolve the fat; in this menstruum fat is easily soluble while hot, though it largely deposits on cooling; the other constituents of milk are almost insoluble. A series of comparative experiments showed that no appreciable difference was found between ether and amyl alcohol. Amyl alcohol is readily volatile at 100° in a current of air.

The method used is as follows: 4 grammes of cream are weighed into a large platinum dish and evaporated in the water-oven; after about one hour the solids-not-fat become adherent to the basin, and by inclining it the fat runs away to the lower portion, and the solids-not-fat are left comparatively free from fat. Under these circumstances drying is complete in about five to six hours. To extract the fat 25 c.c. of amyl alcohol are poured on, and the basin placed for a short time in the water-oven; the amyl alcohol is then carefully decanted; the solution of fat can be separated without loss of solids-not-fat if attention be paid to the slow decantation. This process is repeated about eight or nine times, the basin being usually allowed to stand overnight between the fourth and fifth treatments. The following experiment will show the results obtained with ether and amyl alcohol:

	Weight Taken.	Weight after Drying at 100°.	Weight after Four Washings and Three Hours' Drying.
Ether ...	3·9985 ...	2·898 ...	0·246
Amyl alcohol	3·998 ...	2·894 ...	0·2405

	Weight after Four more Washings and Three Hours' Drying.	Weight after Four more Washings and Three Hours' Drying.
Ether ...	... 0·240 ...	0·2385
Amyl alcohol	... 0·239 . .	0·238

The solids-not-fat are then dried for about three hours at 100° C. and weighed, the fat being found by difference. Determination of the fat by the Werner-Schmid or Leffmann-Beam methods gives results agreeing within the limits of experimental error:

Fat by Difference.	Werner-Schmid.	Leffmann-Beam.
50·60 ...	50·58 ...	50·14
57·16 ...	57·20 ...	57·35 56·95
51·78 ...	— ...	51·89 51·67
56·73 ...	— ...	56·94

The solids-not-fat left in the basin on incineration at dull redness give the percentage of ash.

Clotted cream analysed by the method given above yielded the following results:

	Maximum.	Minimum.	Average.
Total solids	75·50 ...	55·16 ...	66·64
Fat	67·69 ...	44·29 ...	58·22
Ash	1·11 ...	0·44 ...	0·70
Solids-not-fat	11·51 ...	5·68 ...	8·42

The following percentages of water in butter have been found:

	Maximum.	Minimum.	Average.
English fresh butter ...	14·64 ...	11·59 ...	13·05
,, salt ,, ...	15·44 ...	11·50 ...	13·23
French fresh ,, (X)	17·82 ...	14·30 ...	15·73
,, ,, ,, (Y)	16·31 ...	13·08 ...	14·33
,, salt ,, ...	14·84 ...	9·09 ...	12·73
Danish ,, ,, ...	17·10 ...	8·90 ...	12·58

The English butters were examined within twenty-four hours of churning; it is seen that there is no appreciable difference between fresh and salt butter.

The most notable point is the high percentage of water in French fresh butters; these have been divided into two classes according to the district of origin. Of the X samples no less than 40 per cent. contained above 16 per cent. of water, which has been proposed as a limit, the maximum reaching 17·82, while the average is only just below 16 per cent. Only one sample of series Y contained above 16 per cent. of water. Two samples of Danish salt butter contained above 16 per cent. of water; on microscopic examination the appearance described by Storch (ANALYST, vol. xxii., p. 197) as "thick"—*i.e.*, the presence of enormous numbers of small water-globules—was noticed. They did not appear at all watery on casual examination, but seemed to be slightly "overworked"; salt butters containing a lower percentage of water almost invariably look "wet" if a short period after churning has elapsed. The French fresh butters did not appear "thick," but had a tendency to break off short, or, in other words, had a low tensile strength.

The manufacturers of the X butters have evidently worked out a method for retaining a somewhat large percentage of water, without in any way deteriorating the flavour or keeping qualities; seeing that the same thing occurred in 1895, and to a lesser extent in 1896, I believe I am justified in regarding the extra average percentage of water (2 per cent.) as an addition. An interesting and difficult question is raised: Is this addition (which may be safely assumed to be commercially successful) to be regarded as an adulteration, seeing that the essential qualities of the butter (flavour, texture, etc.) are not only retained, but manifest themselves in a high degree? I may add that there was no suspicion of adulteration by margarine; the butyro-refractometer results usually fell between 45° and 46° at 35° C., and never appreciably exceeded 46°.

DISCUSSION.

Mr. BEVAN inquired what was regarded as the particular advantage of amyl alcohol over ether in the determination of fat. Unless absolutely pure, it would contain substances boiling at a high temperature, which he doubted very much would be volatile at 100° C., even in a current of air.

Mr. L. K. BOSELEY said that it was never suggested that amyl alcohol should be used in a Soxhlet, but merely for extracting the fat from quantities of, say, 100 grammes of butter, the advantage being that it could be boiled in a water-bath without loss. Amyl alcohol would extract fat much better at a high temperature than ether would at a low one; and any residual non-volatile substances could be afterwards washed out with ether.

ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

FOODS AND DRUGS ANALYSIS.

A Delicate Test for the Detection of a Yellow Azo Dye used for the Artificial Coloring of Fats, etc. J. F. Geisler. (*Journ. Amer. Chem. Soc.*, 1898, xx., 110-113.)—By the food laws of several of the American States, the addition of an artificial coloring matter to oleomargarine is forbidden. The author states that azo dyes have now largely replaced annatto as the coloring substance, and calls attention to the fact that fuller's earth gives a pink or red colour, with at least one, if not more, of these dyes. The dye can be precipitated from the clarified fat by the earth as a violet-red precipitate, which, when washed with naphtha to remove the fat, and dried, leaves a violet-red powder. This is immediately decolorized on contact with alcohol, the colour reappearing on evaporation of the latter. The coloring matter can be extracted with boiling alcohol, and as thus obtained is insoluble in cold and sparingly soluble in hot water. It dissolves in concentrated sulphuric acid with a yellow colour, which changes to red on the addition of water. Other strong mineral acids also produce a violet or pink colour. In the latter particular it behaves like methyl orange, which, however, when pure does not give the fuller's earth reaction.

The reaction, with the help of the microscope, is exceedingly sensitive ; and whilst 14 grains of the coloring matter per ton of fat give a barely perceptible faint yellow tint, a strong pink tint is obtained on spreading ½ gramme of the sample on a porcelain slab, and adding fuller's earth. When the fat is dissolved in naphtha, and the earth added to the solution, the pink appears as a zone at the edge of the deposited reagent.

It is stated that commercially the yellow azo dye is used in combination with an orange dye, and that the latter does not give this reaction. C. A. M.

The Determination of Antipyrine and of Iodine. J. Bougault. (*Journ. Pharm. Chim.*, 1898, vii., 161-163.)—One gramme of antipyrine combines quantitatively with 1·351 gramme of iodine, and on this fact the author bases the following method of estimating these bodies by means of one another. To 20 c.c. of a solution of 1 gramme of antipyrine in 100 c.c. of 95 per cent. alcohol are added 20 c.c. of an alcoholic solution of mercuric chloride (2·5 grammes in 100 c.c.), and the solution of iodine (1·351 gramme in 100 c.c. alcohol) run in until the liquid becomes faintly yellow. The determination of iodine by means of a solution of antipyrine is made in a similar manner. The reaction is instantaneous, and antipyrine has the advantages over thiosulphate of readily being obtained in a pure and dry state, and of remaining unaltered in solution for an indefinite length of time (*cf.* ANALYST, xxii., 219).

C. A. M.

Opticity of Crystalline Digitalin. A. Petit. (*Bul. gén. de Thérap.*, 1897, ii., 748; through *Chem. Zeit. Rep.*, 1898, 36.)—A 2 per cent. solution of absolutely pure crystallized digitalin in 95 per cent. alcohol has an opticity of +11·6° at 18° C.; a solution of the same strength in chloroform rotates the beam +17·2°. Inasmuch as well-crystallized (German) "digitoxin" has the same melting-point and chemical properties, and also exhibits the same rotatory power both in alcoholic and chloroformic solution, the two bodies are manifestly identical. F. H. L.

The Analysis of Liquorice Mass. A. Mellor. (*Amer. Jour. Pharm.*, 1898, lxx., 36, 137.)—The following process has been adopted by the manufacturers and large consumers in the United States:

Moisture.—Two grammes of the mass are dried in the water-oven until hard. The dry residue is then divided into small pieces and the drying repeated until the weight is constant.

Mineral Matter.—The dried mass is incinerated until the ash is white.

Insoluble Substance.—Five grammes are dissolved in 100 c.c. of water, and after twelve hours the sediment is collected on a weighed filter, cold water being used in rinsing out the beaker. A further deposit occurs in the filtrate after another twelve hours, owing to traces of starch passing through the filter. This is collected on a second weighed filter.

Gummy Matter.—Five grammes are dissolved in 50 c.c of boiling water and 100 c.c. of 95 per cent. alcohol added. The liquid is well stirred, and after standing all night the precipitate is collected on a weighed and dried filter and washed with alcohol (95 per cent.) 2 parts, and water 1 part, until the filtrate is colorless. The residue, dried in the water-oven, gives the combined weight of insoluble substances and gummy matter.

Glycyrrhizin.—The alcoholic filtrate from the above is concentrated to a volume of 30 c.c. and transferred to a weighed beaker with the help of about 20 c.c. of water. The glycyrrhizin is precipitated by adding dilute sulphuric acid (12 drops of strong acid to 5 c.c. of water) with constant stirring. After standing all night, the liquid is poured off through a filter and the glycyrrhizin washed three times with ice-cold water, which is decanted each time. After adding 1 drop of concentrated ammonia-water to neutralize any acid, the beaker is dried in the water-oven to constant weight.

Saccharine Matter.—The liquid decanted from the precipitated glycyrrhizin is neutralized with barium hydrate, the barium sulphate separated by filtration, and the amount of saccharine matter in the filtrate determined with Fehling's solution.

Extractive Substance.—This is taken as the difference between 100 and the other constituents determined as above. C. A. M.

ORGANIC ANALYSIS.

A Volumetric and Gas-volumetric Method for the Determination of Hydroxylamine and Hydrazine. K. A. Hofmann and F. Küspert. (*Berichte*, 1898, xxxi., 64-67.)—This is based on the oxidation of hydroxylamine or hydrazine, with a dilute solution of a vanadium salt in sulphuric acid, the nitrogen evolved being collected and measured, and the partially-reduced solution titrated back with permanganate.

I. $2NH_3O + O = N_2 + 3H_2O$. II. $N_2H_4 + 2O = N_2 + 2H_2O$.

Should other reducing substances be present, a deduction is made based upon the amount of nitrogen calculated to hydroxylamine or hydrazine.

The vanadium solution is prepared by dissolving 5 grammes of ammonium meta-vanadate in 50 c.c. of sulphuric acid kept cool, and diluting to 1 litre. The substance to be analysed is dissolved in dilute sulphuric acid, and the vanadium solution added slowly until a green coloration results. The nitrogen evolved at the ordinary temperature is collected, which requires about twenty minutes. The flask is then warmed at about 60° C. for a few minutes, at the end of which the green colour should still remain, and, finally, the solution is transferred to a porcelain dish and titrated with standard permanganate until there is a permanent rose tint.

The following results were obtained with hydrazine sulphate (15 grammes in 1,000 c.c.). The permanganate solution used contained 1·794 grammes of available oxygen per litre.

Hydrazine Solution. C.c.	Vanadium Solution. C.c.	Permanganate. C.c.	Oxygen consumed.	Nitrogen at 0° C. and 760 mm. C.c.
10	50	20·51	0·03680	
10	50	20·23	0·03630	
10	50	20·20	0·03624	
10	50	19·97	0·03583	19·85 = 22·09 per cent.

The calculated amount of nitrogen = 21·58 per cent., and the results were therefore in accordance with the equation $N_2H_6SO_4 + 2O = N_2 + 2H_2O + H_2SO_4$.

The results obtained with hydroxylamine sulphate, hydroxylamine nitrate, and hydrazine mercury sulphate showed a similar agreement with those required by theory. C. A. M.

The Estimation of Phenylhydrazine. H. Causse. (*Bull. Soc. Chim.*, 1898, xix., 147-149.)—This is based on the fact that arsenic acid oxidizes phenylhydrazine, liberating nitrogen, and leaving phenol, and that a corresponding quantity of the arsenic acid is reduced to arsenious acid.

$$As_2O_5 + C_6H_8N_2 = N_2 + H_2O + C_6H_6O + As_2O_3.$$

The reaction is quantitative in an acetic acid solution, and the arsenious acid produced may be estimated either by titrating the standard arsenic acid solution with uranium before and after the reaction, so as to obtain the amount not reduced, or by titrating the arsenious acid produced with iodine in the presence of sodium bicarbonate ($As_2O_3 + 2I_2 + 2H_2O = 4HI + As_2O_5$).

About 0·20 gramme of phenylhydrazine, or preferably of its hydrochloride, are heated gently under a reflux condenser with 60 c.c. of an arsenic acid solution prepared by dissolving on the water-bath 125 grammes of arsenic acid in a mixture of 450 c.c. of water and 150 grammes of concentrated hydrochloric acid, filtering when cold, and making up to a litre with glacial acetic acid. When the bubbles of gas have ceased, the liquid is boiled for about forty minutes and allowed to cool. After the addition of 200 c.c. of water, a solution of caustic soda (free from sulphides) is added until the liquid is just alkaline. Hydrochloric acid is then added to acid reaction, and, when cold, 60 c.c. of a cold saturated solution of sodium bicarbonate are added and the liquid titrated with decinormal iodine.

If V be the volume of iodine solution required, the quantity of phenylhydrazine is obtained from the formula $V \times 00495 \times 0{\cdot}5454$.

The results given by the author agree well with theory, and it is stated that the method is equally applicable to the analysis of compounds of aldehydes and phenylhydrazine, although if the aldehyde belong to the fatty series it should be eliminated on account of its action on the arsenic acid; if, however, it belong to the aromatic series its presence is without effect, and the estimation can be made on the compound itself. C. A. M.

Examination of Wax with the Refractometer. J. Werder. (*Chem. Zeit.*, 1898, xxii., 38 and 59.)—The author finds that the Zeiss butter refractometer may advantageously be employed in the examination of different kinds of wax, especially when the amount of material at disposal is very limited, and that the indications obtained with it are quite as valuable as in the case of oils and fats. Owing to the high melting-point of the wax, it is necessary to work at a higher temperature than usual, preferably 66° to 72° C., and then to reduce the results to the normal temperature, 40° C. As shown in the annexed table, the figures given by genuine beeswax vary from 42·6° to 45·4°, the great majority of specimens falling between 44° and 45°; and it seems to make little or no difference to the refractive power whether they are tested before or after bleaching. Samples 19 to 24 had previously been examined chemically, and had been rejected on the ground of their abnormal acid and ester numbers, which were as follows:

Number of Sample.		Acid Number.		Ester Number.
19	...	18·48	...	66·64
20	...	127·1	...	13·4
21	...	59·08	...	3·36
22	...	104·7	...	14·3
23	...	41·0	...	57·0
24	...	106·9	...	48·1

No. 24 is a product, called "glanzwachs," obtained by adding some of the mixture of stearic and palmitic acids as used in the manufacture of stearin candles (No. 28) to a genuine wax, this being a form of adulteration commonly employed in Switzerland.

REFRACTIVE POWER OF DIFFERENT KINDS OF WAX.

	Sample.	Temperature of Observation.	Refraction at 40° C.
1.	Bleached, from Egypt	66·0 ...	44·1
2.	,, ,, Turkey	67·0 ...	44·8
3.	,, Moldavia	66·5 ...	44·2
4.	Yellow, Egypt	66·0 ...	42·8
5.	Monte Christo	71·0 ...	44·8
6.	France	67·5 ...	44·1
7.	Savoy	67·0 ...	42·6
8.	California	69·5 ...	45·2
9.	North Africa	71·0 ...	45·0
10.	Massowah	71·5 ...	44·3
11.	Italy } different samples ...	70·0 ...	44·9
12.	,, }	70·0 ...	44·0
13.	,, }	68·5 ...	44·6
14.	Mexico } different samples	69·5 ...	44·2
15.	,, }	67·0 ...	45·3
16.	Syria	69·5 ...	44·2
17.	Casablanca	68·0 ...	45·4
18.	Smyrna	70·0 ...	44·7
19.	Bleached, in chips (professedly genuine) ...	70·5 ...	41·3
20.	White Church candles ,, ...	67·5 ...	32·0
21.	,, ,, ...	68·0 ...	32·5
22.	,, ,, ...	68·5 ...	32·6
23.	Yellow wax, source unknown	66·0 ...	38·3
24.	Wax adulterated with No. 28	65·5 ...	38·8
25.	Paraffin	65·0 ...	22·5
26.	Ceresin	77·0 ...	41·0
27.	Tallow	71·5 ...	48·5
28.	Stearin candle material	70·0 ...	30·0
29.	Carnauba wax	91·0 ...	66·0
30.	Japan wax	71·0 ...	47·0

F. H. L.

On the Estimation of Unsaturated Fatty Acids. E. Twitchell. (*Jour. Soc. Chem. Ind.*, 1897, 1002-1004.)—The author bases a method for separating saturated from unsaturated fatty acids on the fact that the latter combine (in all probability quantitatively) with sulphuric acid, forming addition compounds which are insoluble in petroleum spirit. As it was found that the saturated acids could not be extracted from a concentrated sulphuric acid solution by petroleum spirit, and that the addition of water decomposed the sulpho-fatty acids, experiments were made with sulphuric acid previously diluted, and eventually 85 per cent. was fixed upon as the most satisfactory strength.

The method of separation tentatively adopted is as follows: From 0·5 to 1 gramme of the fatty acids are melted in a stoppered Erlenmeyer flask, the flask chilled in ice-water, 3 c.c. of 85 per cent. sulphuric acid added, and the temperature allowed to rise. As soon as the action commences a clear solution is rapidly obtained, and the flask is again cooled. Fifty c.c. of petroleum spirit are then introduced, the

flask well shaken, the petroleum spirit decanted into a separator-funnel, the flask rinsed out twice with 10 c.c. of petroleum spirit, and the total extract washed with water, the solvent evaporated, and the residue, consisting of saturated fatty acids, dried and weighed.

Crude oleic acid, solidifying at 12° C., when examined in this way, gave a crystalline residue melting at 37° C., and the following results were obtained with the fatty acids from three different samples of oil:

Origin of Fatty Acids.	Solidifying Point, ° C.	Amount used. Gramme.	Petroleum Spirit Extract, per cent.	Melting-point of Saturated Acids, °C.
Lard	40·75	0·7815	42·35	53·5
Cotton-seed-oil ...	30·89	0·6150	32·60	...
Ditto	31·90	0·5875	23·91	53·0

From the melting-points of the residues being somewhat low, the author did not consider the saturated acids thus obtained quite pure, although the impurity must have been slight.

A second extraction of the sulphuric acid solution of the cotton-seed-oil fatty acids, with 50 c.c. of ether, yielded 0·019 gramme of residue which did not solidify at the ordinary temperature, such residue being attributed to the decomposition of a small amount of sulphostearic acid.

The petroleum spirit used must first be tested to see whether any non-volatile substances are produced by treatment with sulphuric acid. If so, it can be purified by being digested for an hour at 100° C., with about half its weight of concentrated sulphuric acid, and then washed and distilled. C. A. M.

Estimation of Phenol in Disinfectants in Presence of Soap. W. Spaltehols. (*Chem. Zeit.*, 1898, xxii., 58.)—In the examination of neutral disinfectants, such as creolin, lysol, and "soluble cresol," where the phenols are not in a state of combination, there is no necessity to add any acid before distillation, as recommended by Fresenius and Makin (ANALYST, xxi., 301), since calcium phenolates are readily decomposed when heated in aqueous solutions. The sample is placed in an iron retort and distilled in a current of steam between 200° and 220° C. until the distillate no longer yields any oily matter. Bodies which contain soaps of oleic acid must not be heated above 210°, lest the latter are decomposed; but should this happen, it will at once be rendered apparent by the presence of a layer of oil floating on the top of the water in the receiver. Alkali-rosin soaps easily resist a temperature of 220°. The distillate consists of phenols alone in the case of lysol; of phenols and tar hydrocarbons in the case of creolin, mixed with the water; and the simplest way of separating them is to extract the whole with benzene, remove the aqueous portion, and estimate the phenols themselves with caustic soda. (Koppeschaar's process is not adapted for the analysis of mixed phenols of unknown composition.) Tried on a number of known products, the author's method has given results usually 0·5 per cent., but occasionally 1·0 per cent., below the theoretical; and it is therefore quite accurate enough for ordinary work.

Lysol and "soluble cresol" contain between 50 and 60 per cent. of phenols; creolin from 0 to 28 per cent., although samples which emulsify well with water seldom have more than 18 per cent. The relative values of the two materials, however, cannot be judged merely by the proportion of phenols found in them, for if a creolin gives an oily precipitate on dilution with water, that portion of the substance is wasted; while, on the other hand, the neutral hydrocarbons present also exhibit distinct germicidal properties. F. H. L.

A Reaction distinguishing between Creosotes and Guaiacols. H. Fonzes-Diacon. (*Bull. Soc. Chim.*, 1898, xix., 191, 192.)—A small quantity of the sample is dissolved in water, 2 or 3 c.c. of a solution of copper sulphate (about 4 per cent.) added, and 1 or 2 c.c. of a 4 per cent. solution of potassium cyanide. An immediate striated precipitate is produced, which, viewed by transmitted light, is emerald-green in the case of creosote, grayish-red with poor guaiacol, and maroon-purple with rich guaiacol. The emerald-green colour changes rapidly to yellow. In this way it is possible to determine whether a product is a creosote containing 12 to 25 per cent., a guaiacol containing 65 to 70 per cent., or a guaiacol with 85 to 90 per cent. of crystallizable guaiacol, without having recourse to a colour scale of typical solutions as in Adrian's colorimetric method (ANALYST, xxii., 162). C. A. M.

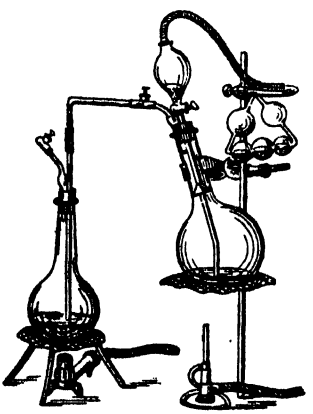
Detection of Halogens in Organic Compounds. P. N. Raikow. (*Chem. Zeit.*, 1898, xxii., 20.)—Some ten years ago Günzburg recommended the use of an alcoholic solution of phloroglucinol and vanillin to detect free hydrochloric acid in the gastric juice; for the reagent gives an intense permanent red colour on warming therewith, although it is unaffected by organic acids. If the same solution is heated with an organic body containing a halogen, only in a few cases is the red colour produced. If the substance is liquid and combustible, it may be mixed with a few drops of the phloroglucinol-vanillin solution in a flat porcelain basin, and the alcohol ignited. As the spirit burns away, the red colour usually develops; but to render the test universally applicable it is better to operate as follows: A piece of porcelain is moistened with the reagent, the solvent allowed to evaporate, and the dried film (which is now colorless) is held over the flame of a spirit-lamp, into which the suspected substance is introduced on the end of a platinum wire. The test is roughly quantitative, for according to the amount of halogen present, more or less of the surface is turned red. If the organic substance is an inflammable gas, it may be set light to, and the porcelain dish held over the flame. F. H. L.

Estimation of Carbon and Oxygen in Organic Bodies by Moist Combustion. J. K. Phelps. (*Zeits. anorg. Chem.*, 1898, xvi., 85.)

Oxidation of Carbon with Potassium Permanganate.—Many organic substances are completely oxidized on warming with sulphuric acid and permanganate, so that the evolved carbon dioxide may be absorbed in barium hydroxide, and determined with iodine and arsenious acid as previously mentioned (ANALYST, xxii., 55). The apparatus consists of a wide-necked 75 c.c. flask fitted with a stoppered funnel and leading-tube for the gas, the latter being expanded into a bulb immediately over the

cork; and a condensing-flask of 250 c.c. capacity provided with an inlet tube reaching to the bottom and an outlet closed by a screw clamp. The two corks are of rubber, and so long as they do not come in contact with the liquids they will not be attacked. The substance is rinsed into the small flask with 10 or 15 c.c. of water; 3 or 5 c.c. more of standardized caustic baryta solution than is necessary to absorb all the gas is introduced into the larger vessel, the pressure reduced by means of a pump to 200 or 225 mm., the organic solution warmed, excess of permanganate run in through the tube funnel, and, finally, 10 c.c. of 1 : 4 sulphuric acid. The mixture is boiled for five minutes, care being taken to maintain some vacuum, while the receiver is well shaken and kept cold in a basin of water; pure air is then allowed to enter through the funnel to restore the atmospheric pressure, and to drive the last traces of carbon dioxide into the baryta. The cork of the large flask is removed, the tubes washed, another rubber cork carrying a funnel and a set of nitrogen bulbs put in its place; the liquid is heated, decinormal iodine added till it becomes permanently red, then cooled again, and the excess of iodine titrated with decinormal arsenious acid. The permanganate solution should be boiled with sulphuric acid until all CO_2 is driven off; the water and acid must also be boiled till free from the gas. Oxalates may be decomposed very smoothly in this way; but in the case of formates and tartrates more than sufficient pure caustic soda (not ammonia) should be added at first to neutralize the acid in the permanganate, and then, after the whole has been raised to the boil, an excess of dilute sulphuric acid run in as before. The process was checked on ammonium oxalate, barium formate, and tartar emetic; the figures show an average error of about 0·2 milligramme in estimating 0·15 to 0·6 gramme of CO_2.

Oxidation of Carbon with Chromic Acid.—Although a concentrated mixture of chromic and sulphuric acids is a much more powerful oxidizing agent than potassium permanganate, there are many organic substances, as Cross, Higgin, and Bevan have pointed out, which yield some carbon monoxide on treatment therewith. The following arrangement ensures complete oxidation without the necessity for passing the gases over red-hot copper oxide. The construction of the apparatus is explained by the annexed cut. The large flask is made of thick glass and holds 1 litre; the cone in the neck is of platinum, and serves to protect the rubber cork from splashes when the vessel is shaken. The separating funnel holds 100 c.c.; the stopcock is well ground in, and moistened with strong metaphosphoric acid. The condensing flask holds 500 c.c. The substance to be analysed is weighed into a thin glass bulb, the aperture thereof sealed, the bulb dropped into the large flask, and an excess of pure potassium bichromate added. A suitable amount of caustic baryta solution is poured into the condenser, the apparatus put together, 10 c.c. of pure sulphuric acid introduced into the generator, and both flasks are boiled till about 2 or 3 c.c. of water have been evaporated from each, and a vacuum is produced. The flames are removed, the two clamps shut, the little bulb is broken by a jerk, and 20 c.c. of strong



sulphuric acid (previously freed from organic matter by heating with a few crystals of bichromate) run in through the funnel. The flask is well shaken, heated to about 105° C. (the maximum temperature permissible if loss of oxygen is to be avoided), water added to dissolve the chromic acid crystals, and the whole is again boiled (without allowing the pressure to exceed that of the atmosphere) for five minutes with constant agitation. By this time any CO will be oxidized; 60 or 70 c.c. of water are introduced, the clamp between the two flasks is opened, and the carbon dioxide is permitted to pass into the condenser, which is kept cold and shaken as before. The contents of the generator are once more heated to the boil, and a steady current of pure air is passed through the apparatus for fifteen minutes. Finally the CO_2 is estimated in the manner already given.

Estimation of Oxygen. — In the above process, if a known amount of pure bichromate is employed, and the excess of chromic acid remaining unreduced after the operation is determined, it is evident that the quantity of oxygen required to burn the carbon may be calculated; and from the total CO_2 recovered, by a simple subtraction, the oxygen in the original body may be deduced. The only special precaution necessary is that the sulphuric acid shall not become too strong, lest it begin to act on the bichromate, causing the evolution of oxygen instead of chlorine in the second distillation. Twenty c.c. of sulphuric acid are used in the C estimation, and the boiling is allowed to proceed quietly. The volume of water added in the final dilution should be adjusted so as to leave 60 or 80 c.c. in the flask when the CO_2 has been driven off. This liquid is brought into a Voit flask connected with a Drexel washing apparatus containing an excess of sodium arsenite of known strength, and a set of nitrogen bulbs filled with dilute caustic soda. It is treated with 35 c.c. of HCl (specific gravity 1·2), boiled in a gentle current of CO_2, which has been washed in a solution of iodine in potassium iodide and then in KI alone, until some 30 or 40 c.c. have distilled off. Sometimes red vapours of chromyl chloride are produced during the boiling; but as this substance is reduced by the arsenious acid, it is a matter of no consequence to the analysis. The latter liquid is acidified with sulphuric acid, made alkaline with potassium carbonate, and titrated with decinormal iodine.

The double method (for C and O) was tested on ammonium oxalate, phthalic acid, pure sugar, paper, tartar emetic, and barium formate. The results are exemplified in a table: they are, especially for the carbon, eminently satisfactory; the error in the oxygen determinations varies from 0·0 to 2·1 milligrammes in 0·1 to 0·5 gramme. Organic substances which are volatile and yet difficult to oxidize cannot be subjected to this moist combustion: ether is easily oxidized to acetic acid, but it cannot be completely burnt up; for although liquid acetic acid is very rapidly attacked by nascent chromic acid, yet in the gaseous state (as it exists owing to the conditions of the reaction) it resists oxidation. Similarly, naphthalene is not amenable to the author's process. F. H. L.

Separation of Ethylene from Benzene in Gas. E. Harbeck and G. Lunge (*Zeits. anorg. Chem.*, 1898, xvi., 26-50.)—In the ordinary analysis of coal or coke-oven gas, it is usual to estimate the "heavy hydrocarbons" by absorption with fuming sulphuric acid or bromine, making no distinction between the benzene and

the ethylene in spite of the fact that these substances are of very different value either for illuminating or heating purposes. The authors have investigated two methods of separating these hydrocarbons, the first depending upon the conversion of ethylene into ethane in presence of hydrogen and platinum black, the second on the nitration of the benzene. A full description of the preliminary experiments, the apparatus necessary, and the final calculations, is given in the original article; but it is too lengthy and replete with detail to be properly abstracted; the following cursory account, however, explains the principles underlying the two processes.

Conversion of Ethylene into Ethane.—A U-shaped tube, about 10 cm. long and 3 mm. in internal diameter, with capillary ends, is filled with 0·5 gramme of platinized asbestos (= 0·11 gramme of Pt), through an aperture in the base, which is afterwards sealed up. It is hung within a glass or metal beaker full of water or copper turnings, according to the temperature at which it is to be employed. A current of pure hydrogen is next passed through it for two hours at 100° C., and two hours in the cold to saturate the platinum black; and the apparatus is then ready for use. In one sample of the gas to be analysed, which must obviously contain an excess of hydrogen, the total amount of ethylene and benzene is determined by absorption in fuming sulphuric acid, the other constituents being estimated in the usual way. A second sample is freed from oxygen with alkaline pyrogallol, and led two or three times over the platinized asbestos at 100° C. The residue is treated with fuming sulphuric acid as before, which now absorbs the benzene only, the difference between the volume taken up before and after condensation thus giving the percentage of ethylene. As 1 volume of ethylene unites with 1 volume of hydrogen to form 1 volume of ethane, the proportion of the former ingredient may be more simply deduced from the contraction; but by operating in this manner it is impossible to determine also the hydrogen and the methane. Unfortunately, the process is not available for those many cases in which carbon monoxide is present in the original gas.

Direct Estimation of the Benzene by Nitration.—The sample of gas is passed through a 10-bulbed tube containing a mixture of equal parts by weight of pure strong sulphuric acid and fuming nitric acid (specific gravity 1·52), which converts the benzene into metadinitrobenzene, and removes the whole of the ethylene. The acid liquor is diluted with ice-cold water, neutralized with caustic soda (also in presence of ice), the bulk of the dinitrobenzene collected on a filter-paper (if there be sufficient of it precipitated to be worth filtering), and washed till free from acid. The filtrate and washings are made up to some convenient volume, and an aliquot portion is extracted twice with ether. The solvent is distilled off, the residue dried in a current of air, dissolved in fresh anhydrous ether, mixed with the rest of the product, the solution filtered, again evaporated, and the solid matter dried at 70° or 80° C. (or over sulphuric acid in vacuo) and weighed. The precipitate thrown down on neutralization is quite pure and without odour; that extracted by means of the ether is apt to be contaminated with mononitrobenzene; but the melting-point of the whole is generally about 86·5° C. (instead of 90° C.).

The reaction is perfectly quantitative; the products formed by the action of the acids on ethylene remain in the aqueous liquid, and do not affect the purity of the dinitrobenzene. Nevertheless, as the process is somewhat tedious and requires a

complicated apparatus, it should only be resorted to for specially important analyses as a check on the former method.

To convert the weight (N) of dry dinitrobenzene in grammes into the percentage by volume (V) of moist benzene vapour in moist gas at the temperature $t°$, and pressure b mm., the following formula may be used: s is the proportion of benzene plus ethylene, W that of the hydrogen in the original gas, and e the tension of aqueous vapour at $t°$ C.

$$V = 98{\cdot}50564 \times \frac{N(1 + 0{\cdot}00367t)(100 - s)}{W(b - \frac{31}{40}e)}.$$

F. H. L.

Use of Lead Carbonate in Analysis. G. Morpurgo. (*Giorn. di Farm. di Trieste*, 1897, ii., 355; through *Chem. Zeit. Rep.*, 1898, 19.)—The author recommends the use of freshly-precipitated lead carbonate in all cases where the acetate is usually employed, *e.g.*, for the removal of coloring-matter, acids, tannins, etc., from complex solutions. The moist carbonate only requires shaking with the liquid, does not dilute it, while the minute trace of lead which passes into solution can be easily removed by a small crystal of sodium sulphate. F. H. L.

Oil of Basil. J. Dupont and Guerlain. (*Bull. Soc. Chim.*, 1898, xix., 151-154.)—Two essences are known by the name of basil—one collected in the South of France and in Germany, having a sweet characteristic odour; the other imported from Reunion, being marked by a strong smell of camphor, which partly masks the characteristic odour. Moreover, the former is lævo-rotatory, the latter dextro-rotatory.

Dumas and Peligot (*Ann. Chim. Phys.*, lvii., 334) extracted from oil of basil a crystalline inodorous product, which they considered to be a hydrate of terebenthine $C_{10}H_{16} + 3H_2O$. The authors, however, have not been able to find any trace of this substance in two samples of the French essence.

The French oil examined by them was an oily yellow liquid with a specific gravity of 0·9154 at 15° C. and a rotation in a 100 mm. tube of $-7{\cdot}40°$. Four-fifths of the oil distilled over between 190° C. and 220° C., and the distillate was further fractionated into two main portions boiling at 195° C. to 200° C. and 205° C. to 215° C. respectively. The former, constituting nearly 60 per cent. of the essence, was an oily liquid, which was identified as linalol ($C_{10}H_{18}O$) by its composition and chemical and physical properties. The fraction boiling at 205° to 215° had the odour, chemical and physical characteristics of *p*-methoxy-allyl-benzene, the chief constituent of oil of tarragon.

A sample of Reunion oil examined by the authors was also found to contain *p*-methoxy-allyl-benzene, but no linalol, a fact confirmed by Bertram and Walbaum (*Arch. der Pharm.*, February, 1897), who found their sample to contain 60 per cent. of that compound and a small quantity of a dextro-rotatory camphor. The results of their analysis of a specimen of German oil showed a close agreement with those obtained by the authors in the case of French oils, and confirmed their conclusions as to the presence of linalol in the European essences and of camphor in the Reunion oil.

C. A. M.

Examination of Rose-Oil. P. N. Raikow. (*Chem. Zeit.*, 1898, xxii., 149.)—The present author is unable to corroborate the figures previously quoted (ANALYST, xxiii., 12) as characteristic of true rose-oil, and he does not consider Dietze's specification of much value. His own results are shown in the subjoined table. The samples are undoubtedly genuine, many of them having been extracted by himself from different varieties of the plants cultivated in different parts of the rose-growing districts. Nos. 1, 2, and 3 are mixed oils, made from red and white roses (as it is the custom to do with marketable specimens); 4 is from red roses alone (*R. centifolia*); 5 from inferior kinds of red flowers; 6 and 7 are the favourite and expensive "green rose-oils"; 8 is from Seraphimoff and Co., of Kazanlik, said to be the same as that examined by Dietze, who gave it an acid number of 1·2 and a saponification number of 9·2. The two samples of geranium-oil are from König and Co., of Leipzig-Plagwitz; the first is called "Ol. Geranii Turkicum rect. alb.," the second "Ol. Geranii Gallic. 1A." The specific gravities of the rose oils were determined at $\frac{27{\cdot}5^\circ\ \text{C.}}{15{\cdot}0^\circ}$, except No 8, which was at $\frac{27{\cdot}5^\circ\ \text{C.}}{17{\cdot}5^\circ}$; those of the geranium-oils were observed both at 27·5° and at 15° C. The optical examination of the rose-oils was carried out in a 100 mm. tube at 25° C.; that of the geranium-oils at 19° C. The solidifying-point is the temperature at which the first crystals of stearoptene were deposited. The acid and saponification numbers are nearly all the mean of two or more tests; the ratio in the last column is that between the acid and the ester number.

ROSE-OILS.

	Specific Gravity.	Solidifying point.	Rotatory Power.	Acid Number.	Saponification Number.	Ester Number.	Ratio.
No. 1	0·8531	22·5	−2° 12′ 15″	1·6	17·7	16·1	1 10·1
No. 2	0·8583	20·5	−2° 6′ 50″	2·3	16·5	14·2	1 6·1
No. 3	—	22·5	−2° 38′ 40″	1·5	16·9	15·4	1 10·2
No. 4	0·8659	18·5	−2° 35′	0·8	13·1	12·3	1 15·4
No. 5	—	22·5	−2° 45′	(2·5)	(16·8)	14·3	1 5·7
No. 6	—	14·5	−1° 43′ 40″	—	17·8	—	—
No. 7	—	27·2	−3° 28′ 30″	2·7	21·1	18·4	1 : 6·8
No. 8	0·845	24·3	−3° 3′ 50″	1·3	10·8	9·5	1 : 7·3

GERANIUM-OILS.

	Specific Gravity.	Rotatory Power.	Acid Number.	Saponification Number.	Ester Number.	Ratio.
Turkish	0·8867 (27·5°) / 0·8960 (15°)	+0° 41′ 20″	1·0	39·6	38·6	1 : 38·6
French	0·8869 (27·5°) / 0·8971 (15°)	−7° 52′	7·7	62·8	55·1	1 : 7·2

F. H. L.

The author concludes that the "constants" relied on by Dietze are not sufficiently precise to allow of the certain detection of adulteration.

On the Precipitation of Proteids. H. Schjerning. (*Zeit. anal. Chem.*, 1897, xxxvi., 643-663.)—The fact that ash-free proteids behave in a different manner from

those containing ash led the author to make experiments on the effect of adding salts to different precipitating reagents. It was found that the addition of various quantities of a 10 per cent. solution of calcium chloride did not interfere with the precipitation of proteid matter by stannous chloride. On the other hand, the precipitates obtained with lead, iron, or aluminium acetates gradually decreased as more of a 10 per cent. solution of calcium acetate was added. From this the conclusion was arrived at that in the case of the tin precipitate a sort of double salt with two basic radicles but only one acid was probably produced.

To determine the influence of salts containing a different acid radicle to that of the precipitating salts a similar series of experiments was made with a 0·4 per cent. solution of disodium phosphate. Up to a certain point the addition favoured the precipitation, but when added in excess had a disturbing effect. The reactions taking place were probably as in the equations:

$$2\text{Alb.Pb.CH}_3\text{COO} + \text{Na}_2\text{HPO}_4 = (\text{Alb.Pb})_2\text{HPO}_4 + 2\text{CH}_3\text{COONa} \text{ or}$$
$$2\text{Alb.Pb.CH}_3\text{COO} + \text{Na}_2\text{HPO}_4 = (\text{Alb})_2\text{Pb} + \text{PbHPO}_4 + 2\text{CH}_3\text{COONa}.$$

In applying this to the method of separating proteids the solutions of the precipitants were of the same strength as given in the *Zeit. anal. Chem.*, xxxv., 286, and whenever the proteid solution contained little or no phosphate the phosphate solution was added in the proportion of 20 c.c. to 6 c.c. of the lead acetate solution or to 0·8 gramme of ferric acetate and 40 c.c. of dilute acetic acid (15 c.c. of 40 per cent. in 1 litre). Care was taken that the proteid solution did not contain in 10 c.c. more nitrogen than corresponded to about 5 c.c. of decinormal acid.

The following table gives the results obtained with various proteid solutions. A cross indicates that the determination could not be made on account of the liquid not filtering clear, or, in the case of the ferric acetate, owing to the iron not completely precipitating on boiling:

	Solution.	Stannous Chloride. Without $CaCl_2$.	Stannous Chloride. With $CaCl_2$.	Lead Acetate. Without Na_2HPO_4.	Lead Acetate. With Na_2HPO_4.	Ferric Acetate. Without Na_2HPO_4.	Ferric Acetate. With Na_2HPO_4.	Normal Uranium Solution.	Precipitation with $MgSO_4$ + Acetic Acid.
Malt I.	100 grammes in a litre	8·5	8·9	18·0	20·6	+	31·9	41·8	19·9
,, II.		8·7	9·9	18·5	20·1	+	33·0	44·8	21·3
Egg-albumen I.	2 grammes in 400 c.c.	+	89·2	90·3	92·3	+	97·2	99·9	96·0
II.*		5·8	91·5	—	—	+	94·2	94·2	93·0
Milk I.	75 c.c. diluted to 500 c.c.	+	82·6	88·8	92·2	+	93·8	92·9	92·1
,, II. ...		+	80·1	88·2	91·9	+	93·0	92·2	92·2
Witte's peptone	2·5 grammes in 500 c.c. ...	1·2	2·7	+	+		59·2	59·2	51·2
Liebig's flesh peptone	5 grammes in 800 c.c. ...		18·9	+	+	+	56·4	53·4	47·2
Liebig's meat extract	5 grammes in 800 c.c. ...	8·4	10·7	+	19·2	+	25·3	36·7	15·1
Diastase (Merck) ...	12 grammes in 500 c.c. ...	15·8	36·9	+	+	+	80·4	85·9	59·7
Urine	50 c.c. diluted to 500 c.c.	0·6	1·4	2·0	2·4	+	3·1	1·9	1·4

* A very old sample.

From comparison with the results obtained with the malt-extract the author considers that there are two kinds of "albumin" present in milk. He divides the precipitated proteids in the following way, and adds, in a subsequent note, that the names "albumin," etc., are only to be regarded as a provisional nomenclature, indicating to some extent the characteristics of the substances.

a = Albumin I. = the tin precipitate.
b = ,, + albumin II. + denuclein = the lead precipitate.
c = ,, + ,, + ,, + propeptone = the iron precipitate.
d = ,, + ,, + ,, + ,, + peptone = the uranium precipitate.
e = ,, + ,, + propeptone = the magnesium sulphate precipitate.

Calculating the results from this, the substances were found to have the following composition:

	Malt Extract.		Egg-Albumin.		Milk.		Witte's Peptone.	Liebig's Peptone.	Liebig's Meat Extract.	Diastase.	Urine.
	I.	II.	I.	II.*	I.	II.					
Albumin I. ...	8·9	9·9	89·2	91·5	82·6	80·1	2·7	13·9	10·7	36·9	1·4
Denuclein ...	11·7	10·2	1·2	1·2	1·7	0·8	8·0	9·2	10·2	20·7	1·7
Albumin II. ...	—	—	1·9	1·5	7·9	11·0	48·5	33·2	0·0	22·8	0·0
Propeptone ...	11·3	12·9	3·9		1·6	1·1			6·1		0·7
Peptone ...	9 9	11·1	2·7	0·0	0·0	0·0	0·0	0·0	11·4	5·5	0·0
Total ...	41·8	44·1	98·8	94·2	93·8	93·0	59·2	56·3	38·4	85·9	3·8

The author remarks that the fact that he finds no true peptone, but only propeptones, in either Witte's or Liebig's peptones is in accordance with the results of König and Bömer; but, on the other hand, he differs from them in finding a large quantity of peptone in Liebig's meat extract (*cf.* ANALYST, xxi., 17). C. A. M.

The Classification of Proteids. A. Wroblewski. (*Berichte*, 1898, xix., 3045-3052.)—The author defines proteids as bodies which on complete decomposition with acids yield as final products ammonia, nitrogenous organic bases (such as lysine, arginine, etc.), and amido acids (such as leucine, tyrosine, etc.). Hence, protamines, which yield no amido acids on decomposition, cannot be classed with the proteids, although closely allied to them. Probably peptones, also, do not comply with the definition, though, for want of more definite knowledge, they may be grouped with their mother-substances, the albumoses.

In the subjoined scheme of classification the proteids are divided into three main groups: I. Albuminous bodies (Eiweissstoffe); II. Compound albuminous bodies (Zusammengesetzte Eiweissstoffe); and III. Albuminoid bodies (Eiweissähnliche Substanzen).

To the first group belong proteids which are closely related to fresh or coagulated white of egg; they contain sulphur in their molecule. *Albumins* are soluble in water. *Globulins* are insoluble in water, but soluble in dilute saline solutions. *Albumins soluble in alcohol* all dissolve in dilute spirit of wine, and many of them in strong spirit. *Albuminates* are formed by the action of alkalies on albumins. They are insoluble in water, but readily soluble in alkalies. *Acid albumins* are produced by the action of acids on albumins, and are soluble in very dilute acids or

alkalies. *Coagulated albumins* are the products of the coagulation of albumins by heat or by enzymes, and are marked by their great insolubility.

The second group comprises proteids whose molecule consists of an albumin group, and another group often of a non-proteid nature. Thus, in *hæmoglobins* there is a coloring-matter group; in *glyco-proteids* a carbohydrate group; in *nucleo-albumins* a nuclëin group; and in *nuclëins* a nucleic acid group.

The third group is subdivided into three classes: (1) Structural substances (Gerüstsubstanzen); (2) Albumoses and peptones; (3) Enzymes.

In the first class are *keratins*, constituents of horn. They contain much sulphur, are only with difficulty attacked by pepsin and trypsin, and on decomposition yield much tyrosin. *Elastins* are contained in the cartilaginous tissues. They are hardly soluble in reagents, contain little sulphur, and on decomposition yield but little tyrosin. *Collagenes* contain very little sulphur, and do not give aromatic amido-acids as decomposition products.

Albumoses and peptones, constituting the second class, have much smaller molecules than the albuminous bodies. By virtue of their toxic properties some of the albumoses are closely related to the enzymes.

The *enzymes*, grouped in the third class, might be further divided in accordance with the conditions of their greatest activity. Thus, some work best in acid solution, others in alkaline solution. Among the former are: pepsin, ptyalin, diastase, invertin, myrosin, and emulsin; whilst representatives of the latter are trypsin, steapsin, and urase.

PROTEIDS.

GROUP I. Albuminous Substances.	GROUP II. Compound Albuminous Substances.	GROUP III. Albuminoid Substances.		
		Class 1. Structural substances. (Gerüstsubstanzen).	*Class* 2.	*Class* 3. Enzymes.
1. Albumins: Egg albumin Serum albumin Lact-albumin Muscle albumin Plant albumin Etc. 2. Globulins: Egg globulin Serum globulin Lacto-globulin Fibrinogen Myosin Plant globulins Vitellin (?) Etc. 3. Albuminous substances soluble in alcohol: chiefly of vegetable origin. 4. Albuminates. 5. Acid albumins: Syntonin and the like. 6. Coagulated albuminous substances: Fibrin Paracasein Coagulated white of egg.	1. Glyco-proteids: Mucins Mucoids. 2. Hæmoglobin. 3. Nucleo-albumins. 4. Caseins: Of cows' milk Of human milk. 5. Nuclëins. 6. Amyloids. 7. Histones (?)	1. Keratins. 2. Ela-tins. 3. Collagenes: Collagene, Glue, and the like.	Albumoses and Peptones.	1. Proteolytic: Pepsin Trypsin Papayotin and the like. 2. Amylolytic: Diastase Invertin and the like. 3. Fat-decomposing enzymes: Steapsin and the like. 4. Glucoside decomposing enzymes. 5. Amide decomposing enzymes: Urase and the like. 6. Coagulating enzymes and the like: Rennet and the like.

C. A. M.

Taka Diastase. J. Takamine. (*Amer. Journ. Pharm.*, 1898, lxx., 137-141.)—In Japan and other Asiatic countries certain fungi are used for the production of diastase. That used in Japan belongs to the genus *Aspergillus*, and is termed *Moyashi*. This is specially cultivated on sterilized wheat bran or other suitable material, and when mature is dried, and the spores separated by shaking or sifting; the product thus obtained is called *Taka-moyashi*, and can be preserved indefinitely. For the manufacture of diastase for commercial purposes, wheat bran is moistened with water, steamed, and, after cooling to below 40° C., is mixed with a little Taka-moyashi, and spread in a layer in a room similar to a malt-floor, where the temperature is maintained at about 25° C., and the humidity at about 80 per cent. Within forty to fifty hours the diastatic power of the mass reaches its maximum, and further growth is checked. The mass is known as *Taka-Koji*, and can be used in the green or dried state. The diastase it contains is soluble in water, and an aqueous extract of the Taka-Koji, concentrated in vacuo to a syrup, has from eight to ten times the diastatic power of malt extract of similar consistency.

The diastase can be precipitated from this aqueous extract by the addition of alcohol, and when separated by means of centrifugal force and air dried is a non-hygroscopic yellowish-white powder, readily soluble in water, and capable of converting over one hundred times its weight of starch in ten minutes. By further purification by re-precipitation or otherwise, its diastatic power, which is exceedingly stable, can be still further increased. C. A. M.

A Simple and Accurate Method of Testing Diastatic Substances. J. Takamine. (*Amer. Journ. Pharm.*, 1898, lxx., 141-143.)—This is based on the great stability of Taka-diastase (see preceding abstract), which does not lose its diastatic power with keeping, as the author finds to be the case in the diastase isolated from malt.

The exact diastatic capacity of a quantity of Taka-diastase is determined once for all by Lintner's or Junk's method (ANALYST, xxi., 122), and that of any substance under examination compared with the standardized sample, and expressed in any terms desired.

Eight glass cylinders, holding about 150 c.c., are placed in water warmed to about 40° C., and into each is poured 100 c.c. of 5 per cent. starch paste. In the first glass is placed 1 c.c. of the saliva or other liquid to be tested, whilst the other seven cylinders receive successively increasing quantities of a freshly-prepared 1 per cent. solution of the standard Taka-diastase, commencing with 1 c.c. in the second cylinder, and ending with 7 c.c. in the eighth. The contents are stirred until the starch becomes liquid, and a drop from each is then removed to a white tile, where it is mixed with 1 drop of a solution of iodine, prepared by dissolving 1 gramme of iodine and 2 grammes of potassium iodide in a little water, and making up to 120 c.c. The drops, when spread out on the tile, with the finger form a colorimetric scale, ranging from blue to purple and reddish-brown, and the colour given by the substance in the first tube is readily matched. C. A. M.

INORGANIC ANALYSIS.

Estimation of Tin in Stannic Salts. **A. Fraenkel and J. Fasal.** (*Mitth. k.k. Tech. Gew. Mus.*, 1897, 227; through *Chem. Zeit. Rep.*, 1898, 11.)—An amount of the stannic salt containing 0·2 to 0·4 gramme of tin is treated with a few drops of strong hydrochloric acid, and 0·5 to 1 gramme of aluminium wire is added. The whole is warmed gently for about half an hour, or until hydrogen is given off briskly, and a fresh piece of wire does not become coated with tin, thus showing that decomposition is complete. Ten c.c. of strong hydrochloric acid are next introduced, and the flask is heated for fifteen minutes till all the metal is dissolved and no more gas is evolved. The solution of stannous chloride is then mixed with Rochelle salt, made faintly alkaline with sodium bicarbonate, and titrated with iodine; or if the greater part of the free acid only is neutralized, Fraenkel's potassium bichromate method may be adopted. The results are slightly too low. F. H. L.

Separation of Beryllium from Aluminium. **F. S. Havens.** (*Zeits. anorg. Chem.*, 1898, xvi., 15.)—This process is identical in principle with that already described by Gooch and Havens for the separation of iron from aluminium by means of ether and hydrochloric acid (ANALYST, xxii., 194); but the details of the operation have been slightly modified, and it is now preferably carried out in the following manner: Fifteen c.c. of the aqueous solution of the mixed chlorides of aluminium and beryllium, containing about 0·2 gramme of the corresponding oxides, are placed in a covered platinum basin, and suspended within a larger vessel through which a current of cold water is constantly passing. The liquid is saturated at 15° C. with gaseous hydrochloric acid, 15 c.c. of ether are added, and the whole is saturated with gas once more. The insoluble aluminium chloride is collected and treated exactly as before; the filtrate, which is allowed to run directly into a platinum crucible, is slowly and cautiously evaporated till all free acid is driven off, then mixed with a little nitric acid to convert it into nitrate, evaporated again, and finally ignited, first at a low temperature and afterwards over the blowpipe. The author states that if the beryllium chloride contains no free HCl when the nitric acid is dropped in, the platinum will not be attacked; but the residue must not be heated more than necessary, lest part of the chloride volatilise. The beryllium can also be determined by removing the excess of HCl and then precipitating with ammonia. The results are sufficiently accurate: calculated as Al_2O_3, the aluminium is from 0·1 to 0·8 per cent. too low; the BeO from 0·4 to 0·8 per cent. too high.

F. H. L.

Use of Metallic Sodium, Magnesium and Aluminium in Blowpipe Analysis. **W. Hempel.** (*Zeits. anorg. Chem.*, 1898, xvi., 22.)—The utility of ordinary blowpipe analysis is somewhat restricted by the difficulty of exposing the substance under examination to a satisfactory reducing flame. This may be overcome in the following manner. A very small, clean fragment of sodium (the size of a grain of millet) is laid on a piece of filter-paper about 4 square centimetres in area, and pressed out with an oily knife until it is quite thin and pliable. The

powdered sample is placed in the middle, and the whole rolled up into a cylinder so that the paper forms a double layer all round. The cylinder is wrapped round with a close spiral of iron binding-wire, the excess of paper cut off, and the cartridge approached to the edge of a Bunsen giving a reducing flame till the paper catches fire. Immediately the reaction is over, the wire is thrust into the centre of the flame to protect the mass from oxidation, and gradually lowered towards the orifice of the burner until it is cooled by the current of gas. The residue is shaken out of the wire and extracted with water in an agate mortar to dissolve the caustic soda. When this has been removed, the insoluble portion can be examined as usual, any reducible metals which the original body contained being present in considerable amount. Silicon and boron will also be found in the elemental state mixed with some carbon from the paper; the residue should then be treated with hydrochloric acid, washed with water, ignited on foil till the carbon is burnt off, when a sufficient proportion of the silicon and boron will still be left unoxidized to respond to the ordinary tests.

The metallic sodium should be kept in an air-tight bottle rather than under petroleum; and if care be taken not to employ too much of it, there is no danger of an explosion. If it be desired to recover a larger quantity of the reduced matter, the mixture may be diluted with common salt, and the reaction effected in an iron crucible. (Silicates may quickly be opened up in this way.)

As the presence of sodium salts hinders the recognition of other metals by their flame tests, a further portion of the sample may be mixed with powdered aluminium or magnesium, wrapped in paper and treated as above; but owing to the infusibility of the earths the reduction is not so complete as with sodium. F. H. L.

The Analysis of Crude Sodium Sulphide. F. Jean. (*Jour. Pharm. Chim.*, 1898, vii., 170-172.)—Ten grammes of the sulphide rapidly broken into large pieces, in order to obtain an average sample, are dissolved in a little water, and the solution after removing the insoluble matter by filtration, made up to a litre. Ten c.c. of this solution are titrated with decinormal iodine with starch as indicator. The volume used corresponds to the sulphide, sulphur in excess, and thiosulphate.

To a second portion of 10 c.c. a solution of ammonium sulphate (6·7 grammes per litre), is added, the same volume being taken as was used of the iodine solution in the preceding titration. After the addition of 30 c.c. of water the liquid is distilled, and the ammonia liberated by the sodium monosulphide received in 20 c.c. of decinormal acid. This is subsequently boiled to remove sulphuretted hydrogen, and titrated with decinormal alkali with turmeric as indicator. Each c.c. of decinormal acid neutralized by the ammonia corresponds to 0·0039 gramme of sodium monosulphide.

The residual liquid in the distilling-flask is titrated with decinormal iodine when cold, each c.c. used corresponding to 0·0079 gramme of sodium thiosulphate. The difference between the amount of iodine solution required here and that used in the first titration corresponds with the sulphur of the sulphides and polysulphides, which may be expressed as sodium monosulphide by multiplying the number of c.c. by 0·0039. The quantity thus calculated is always in excesss of that obtained by the

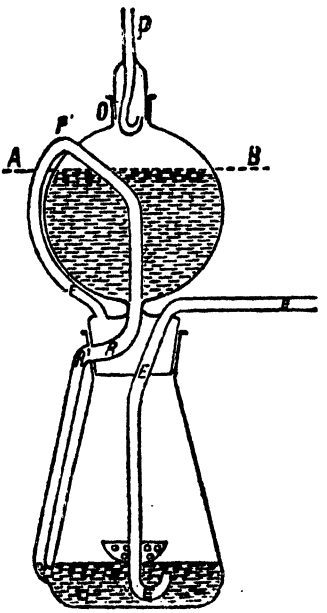
distillation with ammonium sulphate, owing to the excess of sulphur in the polysulphides being included in the monosulphide. This excess is therefore calculated into sulphur (100 of sodium sulphide = 41 sulphur).

If the sample contained only monosulphide the results obtained by the distillation with ammonium sulphate and by the titration with iodine would agree. The result obtained by titrating the sulphide with an ammoniacal solution of zinc is the same as that obtained in the iodine titration (making a deduction for the thiosulphate) since the sulphur of the polysulphides acts upon the zinc like the monosulphide.

In the course of their experiments on this method the authors have established the following facts: (1) When a solution of sodium monosulphide is boiled with an excess of ammonia, part of the sulphur is volatilized in the form of ammonium sulphide; and (2) when a solution of sodium thiosulphate is boiled with an excess of ammonium sulphate there is a liberation of ammonium sulphide and a deposit of sulphur; but this decomposition does not take place in an alkaline liquid, or under the conditions described above. C. A. M.

APPARATUS.

Apparatus for the Valuation of Manganese Peroxide. M. Nothomb. (*Chem. Zeit.*, 1898, xxii., 80.)—This apparatus may either be used for the purpose indicated or for the estimation of carbon dioxide. It is comparatively small, and when fully charged weighs only about 80 grammes. The glass stopper of the flask serves also as a stopcock, opening or closing communication between R and R^1 according as it is revolved in the neck of the flask. The latter contains a small quantity of dilute sulphuric acid, but not enough to touch the perforated receptacle carried by the tube E. Into the acid is put a weighed amount of manganese peroxide, and a few crystals of oxalic acid are laid in the basket. The stopper is then inserted in such a position that R and R^1 are not connected, strong sulphuric acid is poured into the bulb to the height shown, and the whole weighed. Connection is next made between R and R^1 till the oxalic acid is covered with liquid; and when the reaction has ceased, dry air is blown in through E to drive out the carbon dioxide.



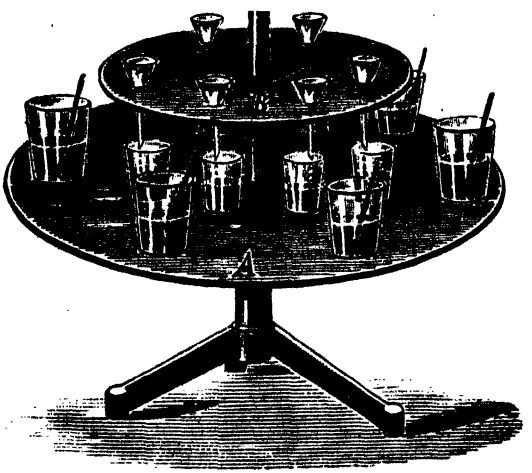
The lower part of the tube F^1 inside the bulb is of course not joined to R, as might be imagined from the illustration given herewith. F. H. L.

A New Measuring Pipette. O. Bleier. (*Chem. Zeit.*, 1898, xxii., 59.)—With reference to the illustration of his pipette on p. 55 of this volume, the author notes that for convenience sake the distance between each bulb should be a trifle greater

than there shown—in fact, that each constriction should be a short tube—as otherwise, although the capacity of the apparatus between each graduation may easily be determined, it will probably work out to some uneven number of cc.s, thus depriving the pipette of much of its practical simplicity.

A modified form of the arrangement, which will be found very useful, especially for measuring large volumes of water, as in diluting liquids or making up standard solutions, may be prepared by constructing the bulbs of different sizes. For instance, the graduated portion *a*—*b* may contain 25 c.c. ; *b*—*c*, 25 c.c. ; *c*—*d*, 50 c.c. ; *d*—*e* 100 c.c. ; *c*—*f*, 200 c.c. ; so that by suitable manipulation any quantity of water can be measured with perfect accuracy. In this manner the use of graduated flasks, which are of necessity marked high up in the neck, can be avoided ; and a standard solution may be prepared in a much larger vessel by weighing the solid, calculating the proper amount of water, and adding it by means of the measuring burette, when, as the vessel need only be half full, the liquid can be shaken satisfactorily till solution is effected.

F. H. L.



A Convenient Filter-stand. H. Faber. (*Chem. Zeit.*, 1898, xxii., 39.)—The construction of this stand is sufficiently explained by the diagram. The tables A and B are either fastened rigidly together so that they revolve simultaneously on the vertical axis C, or B may be carried by a shoulder on the socket rising from A, in order that by the insertion of a suitable number of washers underneath B, the distance between the two tables may be adjusted to suit different sized funnels and beakers.

F. H. L.

An Improved Asbestos Filter. A. Goske. (*Chem. Zeit.*, 1898, xxii., 12.)—The object of this improvement is to reduce the amount of asbestos necessary to ensure efficient filtration, so that while the filtrate is perfectly clear, yet the operation is not unduly prolonged. As will be seen in the accompanying cut, the hollow glass ball has two small holes in its upper part. It is covered with a layer of dry asbestos fibre about 5 mm. thick, then connected with the pump, and a sufficient quantity of asbestos powder previously suspended in water is poured over it in the same way as is done in preparing a Gooch crucible.



F. H. L.

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JAPANESE WOOD-OIL.

By J. H. B. Jenkins.

(*Read at the Meeting, March* 16, 1898.)

Japanese wood-oil (Chinese wood-oil, Tung-oil) is still so little known generally, that the results of the examination of a sample which recently came into the author's hands may be of interest. It is an oil of considerable viscosity, high density, and remarkable drying properties, and is produced from the nuts of the wood-oil tree (*Aleurites Cordata*) in Japan and China, where it is used in large quantities in painting and calking, and for many other industrial purposes. It is said to be extensively used in native medical remedies in virtue of its purgative, emetic, and very poisonous properties when freshly prepared.

For comparison with the results obtained from this sample, others are collated from an earlier sample examined by the author a year ago. Most of these latter results have already appeared in the *Jour. Soc. Chem. Ind.*, 1897, 193 and 195.* The following other references to work done on the oil may be useful: Cloëz (*Bull. Soc. Chim.*, xxvi., 286, and xxviii., 23); Davies and Holmes (*Phar. Jour.*, 1885, 634 and 636); Deering (*Imp. Inst. Jour.*, August, 1896, p. 303); De Negri and Sburlati (*Sc. Mon.*, September, 1897, p. 680).

	Present sample.	Earlier sample.
Specific gravity at 60° F.	0·9343	0·9385
Insoluble fatty acids	96·0%	96·4%
Unsaponifiable matter	0·63%	0·44%
Glycerin	10·6%	10·4%
Free fatty acids (calc. as oleic)	1·83%	3·84%
Viscosity at 60° F. (water takes 28 secs.) ...	858 secs.	1433 secs.
Solidifying point	below − 17° C.	below − 17° C.
Bromo-thermal rise (= $x°$)	21·4° C.	23·4° C.
Calc. iodine value ($x° \times 5·7$)	122·0	133·4
Calc. iodine value ($x° \times 7·0$)	149·8	163·8

* In the Analyst for February, 1898, p. 43, there is an abstract relating to Japanese wood-oil taken from German periodicals. With the exception of a couple of interpolations and errors, the particulars are derived wholly from the author's paper in the *Jour. Soc. Chem. Ind.* above referred to.—J. H. B. J.

	Present sample.	Earlier sample.
Iodine value (Hübl)	... 149·7	165·7
Saponification value (Mgms. KHO)	... 192	194
Specific temperature reaction ...	... 298	330
The insoluble fatty acids gave:		
Melting point	... 30–31° C.	37° C.
Bromo-thermal rise ($= x°$) ...	... 21·0° C.	22·1° C.
Calc. iodine value ($x° \times 5·7$) ...	... 119·7	126·0
Calc. iodine value ($x° \times 7·0$) ...	... 147·0	154·7
Iodine value (Hübl)	... 144·1	150·1

Viscosity.—The viscosity was taken with Redwood's instrument, through which the same quantity of water (50 c.c.) flows in 28 secs., and averages pure rape-oil in 470 secs., at 60° F.

Specific Temperature Reaction (Maumené Test).—The peculiar property of the oil of solidifying in contact with sulphuric acid makes it necessary to largely dilute it before this test is applied. When the acid is added to a solution of 10 grammes of Japanese wood-oil in 40 grammes of diluent, it becomes for a time semi-solid, but soon thins again and permits the maximum temperature to be recorded. A thin mineral oil was first tried as diluent, and an allowance made for the fraction of heat due to it. Mineral oils, however, heat very slowly as compared with fatty oils on treatment with the acid, and such a correction was open to suspicion. For this reason olive-oil was adopted instead, and, although the correction-figure was larger, it was thought to be more satisfactory. The figures given above are calculated from experiments made on 10 grammes of sample diluted with 40 grammes of olive-oil.

Valenta's Test.—The temperature of turbidity with glacial acetic acid was 44° C. The earlier sample gave 47° C.

Elaïdin Test.—In this test the oil darkened considerably. Examined after twenty-four hours, there was an oily upper layer on a more solid portion on the bottom; when stirred up the whole would slowly flow.

Becchi's Argentic Nitrate Test.—A quarter hour's heating with the reagents produced no appreciable deepening of colour. This is different from the earlier sample, which gave a deep reddish-brown coloration.

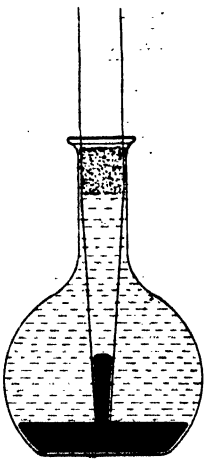
Halphen's Test (*Revue de Chim. Ind.*, February, 1898).—In this test the presence of cotton-seed oil is indicated by the production of a red coloration on heating the suspected oil with amyl alcohol and CS_2 containing some dissolved sulphur. Each of the samples of Japanese wood-oil when tested thus gave negative results. The fact that one of the samples gave the Becchi reaction strongly, but no coloration with the Halphen test, is interesting.

Oxidizing and Drying Properties.—Four grammes of the oil were exposed in the boiling-water oven in a shallow porcelain dish 7 cm. diameter, and weighed after successive hour's exposures. At first there was a slight loss, probably due to moisture in the oil; the average gain of weight during the first eight hours' exposure was 0·26 per cent. per hour. In six hours the surface was entirely covered with a crinkled skin. The earlier sample, under these conditions, gained during four hours exposure at the average rate of 0·36 per cent. per hour, and was covered with a crinkled skin in two hours. A sample of linseed-oil similarly exposed had no sign of

any skin on the surface at the end of eight hours, and the average gain per hour was 0·10 per cent.

Optical Properties.—The sample did not cause any rotation of polarized light. It had very high refractive power at 19° C.; and with the sodium light, its refractive index was 1·503.

Polymerization on Heating.—The oil was heated out of contact with air. The apparatus used (*vide* sketch) was a small flask into the neck of which a glass tube was ground. The tapering lower end of this tube reached below the surface of some mercury at the bottom of the flask. The rest of the flask was completely filled with the oil. During the expansion of the oil a little of the mercury is forced up the central glass tube, but air is rigidly excluded from the oil during its exposure to the heat. The flask was heated in a glass air-bath. The oil was in this way heated first for two hours at 200° C. but remained liquid, then the temperature was raised to 250° C. and kept at that for another couple of hours, during which time the oil thickened and the minute bubbles of gas, which had been arising through the oil for some time, were now held entrapped *en route*. When cold, the oil was found to have been converted into a sticky elastic jelly. The earlier sample of Japanese wood-oil, when similarly treated, yielded a hard, dry, elastic solid. There was no darkening of colour in either case and the products remained clear. There was a decrease of solubility of the oil in all ordinary oil-solvents in proportion as this polymerization change had taken place. This peculiar property was noticed by Cloëz, who also found that exposure to direct sunlight, without any heating, was sufficient to solidify the oil in a few days. Possibly the great difference in viscosity which is noticed in the two samples may be due in some degree to a partial polymerization of one of the samples.



Action of Bromine and Iodine.—The iodine value of the oil was determined by the Hübl method, and the bromo-thermal test applied in the way laid down by Hehner and Mitchell (ANALYST, July, 1895). A year ago these two tests were used comparatively by the author in the examination of a variety of fatty oils, including Japanese wood-oil. It was then found that the remarkable constancy of ratio between the temperature rise on bromine treatment and the iodine value which exists for fatty oils, did not extend to the Japanese wood-oil. Thus, whilst the factor 5·7 applied to the thermal rise gave the iodine value for all the other fatty oils examined (excluding "blown" oils), the factor 7·0 had to be applied for the Japanese wood-oil. It is found that this peculiarity exists also with the present sample of the oil, as well as with the fatty acids from each. (By an error of calculation, it has been stated, *Jour. Soc. Chem. Ind.*, 1897, 193, that this anomaly disappears when the fatty acids are examined; this, however, is not so.) The fact that the heating on bromine treatment is not so great as one would expect from the iodine value suggests that the action of the bromine on the oil is not so extensive as that of the Hübl reagent; but this is hardly so, as the bromine *absorption* value of the oil is not low as compared

with the iodine value. The cause will probably be found in a peculiarity of chemical structure of the fatty acids.

In trying to get some light on the anomaly, the author tried the effect of a chloroformic solution of iodine on the oil. A couple of grammes of the oil were dissolved in 5 c.c. of chloroform, and 5 c.c. of a saturated solution of iodine in chloroform were added with agitation, when the whole was rapidly converted in a jelly, the stiffness of which was proportional to the amount of oil taken. The amount of iodine in the 5 c.c. of chloroform is quite inadequate to saturate the oil. In the earlier sample of Japanese wood-oil 1 gramme of the oil was sufficient to produce a jelly in a couple of minutes, but the present sample is not so sensitive; 2 grammes of it produce a jelly in a couple of hours, and a solution of 4 grammes instantly jellifies on treatment.

This peculiarity has not been noticed with any other fatty oil. A solution of iodine in other solvents produces the same effect. If a saturated solution of iodine in $CHCl_3$ or CS_2 be dropped upon the oil, it immediately solidifies it. Bromine, whether in solution or otherwise, has no similar action on the oil.

The melting-point of the fatty acids from the later sample was not very definite. The solidified fatty acids have a peculiar wavy or rosette-like appearance.

The author is indebted to Messrs. Horner and Sons, of Mitre Square, Aldgate, for the later sample.

Discussion.

The President having invited discussion:

Mr. Hehner said that, owing to the courtesy of Mr. Jenkins, he had been able to make some experiments on a small sample of this oil. He had independently found the remarkable gelatinization referred to, which was a most characteristic property of this oil. This oil was quite different in constitution from any ordinary oil which gave figures of this kind. From the high iodine number it was quite plain that the oil must contain a highly unsaturated acid, in this respect approaching linseed-oil. Linseed-oil formed a hexabromide which was exceedingly insoluble, a proof that linolenic acid was present, and one would expect that this Japanese wood-oil, from its high iodine number, also contained a considerable quantity of linolenic acid. But absolutely no insoluble hexabromide was formed, showing that it must contain a different unsaturated acid from any of those known at the present time. The figures for rise of temperature in the bromine absorption test really applied only to ordinary oils, and he hardly considered this an ordinary oil as hitherto known to chemists. The factor, therefore, failed; but it failed also in every other unsaturated compound in which the acid was other than oleic, linolic, or linolenic acid. This was another proof that Japanese wood-oil contained an acid which was not one of the generally known unsaturated acids.

A TYPICAL NORTH-EAST LANCASHIRE RIVER.

By F. R. O'Shaughnessy.

(*Read at the Meeting, March* 16, 1898.)

Some time ago I had occasion to examine the water of the river Calder, a stream which flows through Burnley. The results of the analyses themselves are only what one would expect to find in a polluted water; but when the series is considered collectively, and in conjunction with the nature of the pollution taking place at different points along the stream, a few interesting points present themselves. Acting on the suggestion of our President at the last meeting, I thought that these points might not prove uninteresting to the members of the Society.

The stream rises on a moor, and the condition of the water here probably coincides with that of the Padiham drinking-water, the analysis of which is contained in sample No. 1 of the table. It flows for some distance through the open country, and then passes through the town of Colne. There there are a number of cotton factories, skin-dressing establishments, etc., and just below the town is the sewage-farm. About half a mile further on the Pendle water, which nearly equals the main stream in bulk, enters. Two miles farther on the stream passes the sewage-farms of Nelson and Brierfield. Lower down still on the bank Burnley sewage-farm lies, and a branch of the river which flows through Burnley enters. A couple of miles more and the stream runs through Padiham, a small town of about 15,000 inhabitants. In the centre of Padiham a brook enters, which consists largely of the refuse from a print-works and a large chemical works. Below Padiham another smaller brook enters similar to the last. A mile or so lower down there are two sewage-farms and a paper-mill, with some settling-tanks for purifying the refuse. The river then flows on through open fields, and receives the effluent from still another sewage-farm through the medium of one tributary, and the refuse from a print-works through a second tributary. Finally it disgorges itself in the Ribble at Thacking Hall.

In the table of analyses the Padiham drinking-water, which appears under No. 1, was analysed in order to give an idea of what a typical good water of the district was like.

Sample No. 2 was taken at a point on the moors about a mile or so from the source of the branch of the river selected. There the dark colour is due to peaty matter, the "total solids" are low, and the "loss on ignition" what one would expect in a peaty water. The "suspended matter" is very low, as is also its loss on ignition, showing that it probably is all derived from the bed of the stream. The lime, chlorine, and sulphuric acid are low, whilst the "oxygen absorption" and "free" and "albuminoid ammonias" are fairly high.

Sample No. 3 was taken below the first large town, viz., Colne, and gets the benefit of the effluents from two sewage-farms. There is a general rise in all the constituents, especially in the free and albuminoid ammonias. We note, however, a fall in the oxygen absorption, and, curiously enough, this corresponds with the trace of nitric acid found in this sample, which shows that oxidation must have taken place.

Sample No. 4 was taken at a point below the towns of Nelson and Brierfield, and the river above this point receives the effluents from their respective sewage-works as well as the two previously mentioned. There we note the immense rise in free ammonia, whilst the albuminoid ammonia falls slightly and the chlorine considerably. This latter fact is due to the entrance of the Pendle water, which is in fairly good condition.

Sample No. 5 was taken below Burnley sewage-farm, and the point where the branch of the stream which flows through Burnley enters. Here the oxygen absorption and free and albuminoid ammonia reach a terrible altitude. In the first four samples we note that the ratio between the loss on ignition of the total solids to the total solids is, roughly, about one-third. But in sample No. 5 this ratio falls considerably. This is in all probability due to pollution from manufacturing sources in the town of Burnley.

Between the points where samples No. 5 and No. 6 were taken there are no sewage-farms. At the same time, there is a considerable amount of water entering the river from brooks and small streams, and the diluting effect of this water is seen in the immense fall in free ammonia. On the other hand, these brooks, etc., consist largely of refuse from print works and chemical works, and their effect is seen in the rise in chlorine and sulphuric and total solids.

The whole of the samples from No. 2 to No. 6 were taken in succession on the same day. Sample No. 7 was not taken till the morning following, and hence is not strictly comparable with the other samples. Between the points where No. 6 and No. 7 were taken there are two sewage-farms and a paper-mill. We see that everything has fallen off but the free ammonia and sulphuric acid. The rise in the former is undoubtedly due to the effluents from the sewage-works. The latter is probably due in part to manufacturing sources, pollution from which, I have reason for believing, takes place pretty heavily during the night.

If we examine the ratio between the loss on ignition of the suspended matter and the suspended matter itself, the steady rise in this ratio as we descend the stream is very striking.

As an appended note to this paper, I would like to draw attention to the use of salicylic acid as a means of preserving a standard solution of sodium thiosulphate, a solution which is much used in water analysis. The figures in Tables I. and II. were obtained by Messrs. Richmond, Boseley and myself in our ordinary routine work by titrating the thiosulphate against 10 c.c. of standard permanganate.

II.

Before addition of salicylic acid:

13 vii.	... 14·6 c.c.	...	
22 vii.	... 14·5 c.c.	9	days
22 viii.	... 15·75 c.c.	40	,,
7 ix.	... 20·20 c.c.	55	,,
9 ix.	... 21·00 c.c.	58	,,
11 ix.	... 23·65 c.c.	60	,,
22 ix.	... 49·00 c.c.	73	,,

I.

After addition of salicylic acid:

25 ix. 97	... 13·95 c.c.	...	
27 ix. 97	... 13·95 c.c.	... 2	days
29 ix. 97	... 14·15 c.c.	... 4	,,
2 x. 97	... 13·95 c.c.	... 7	,,
2 x. 97	... 13·90 c.c.	... 7	,,
11 x. 97	... 13·95 c.c.	... 16	,,
25 x. 97	... 13·80 c.c.	... 30	,,
28 x. 97	... 13·90 c.c.	... 33	,,
28 ii. 98	... 13·90 c.c.	... 156	,,

A Typical River.

Results stated in parts per 100,000.

No. of Sample.	1.	2.	3.	4.	5.	6.	7.
Colour	Almost none	Dark yellow	Faint yellow	Yellow	Yellow	Yellow	Yellow
Smell	Earthy	Faintly earthy	Foul	Foul	Foul	Foul	Foul
Total solids ...	9·10	14·30	31·40	32·20	32·10	36·80	35·00
Loss on ignition	3·00	4·30	8·90	11·20	7·50	10·40	9·50
Suspended matter	—	0·81	1·34	2·89	2·34	1·93	1·39
Loss on ignition	—	0·06	0·97	1·78	1·79	1·52	1·03
Lime (CaO) ...	1·04	3·72	4·88	4·68	4·82	5·60	5·00
Chlorine ...	1·00	1·20	3·40	2·30	2·70	4·40	3·50
Sulphuric acid (SO_3)	0·89	1·39	3·56	3·95	4·67	6·03	6·71
Nitric acid (N_2O_5)	None	None	0·50	None	None	None	None
Oxygen absorbed	0·098	0·333	0·322	0·377	0·414	0·496	0·381
Free ammonia ...	0·006	0·030	0·163	0·472	0·784	0·404	0·462
Albuminoid ammonia ...	0·006	0·015	0·026	0·022	0·040	0·044	0·041

A NEW FORM OF CONDENSER.

By Cecil H. Cribb, B.Sc., F.I.C.

(*Read at the Meeting, March* 16, 1898.)

The form of condenser with which Liebig's name is inseparably connected has served its purpose so well and has been in use for so long that it seems almost sacrilege to attack such a venerable institution. There can be no doubt, however, that theoretically it is most inefficient in proportion to its size, and that practically, in addition to being very cumbrous and heavy, it is in many other ways extremely inconvenient.

A number of other forms have from time to time been introduced, but up to the present not one of them has been at all largely adopted.

With regard to the efficiency of the Liebig condenser, the chemist whose laboratory operations are generally conducted on a small scale has but little reason to trouble. Provided condensation be complete, the actual amount of cooling water used is of little importance, but the absurd size of the present apparatus is a matter much more worthy of consideration. The great amount of bench-room taken up by condensers, and the expense involved in laying on water over large areas, renders it desirable that the space given up to distillation purposes should be as small as is conveniently possible. Any reduction in the size of the condensers must, to be of service, be accompanied by a corresponding increase in efficiency.

A theoretically perfect condenser should present to the vapour a cooled surface as large as possible, while the space into which the vapour has to pass should be as

small as possible consistent with its offering only a very low resistance to the passage of vapour into it. In any case, whatever its absolute or relative size may be, it should be of such form and disposition that the stream of incoming vapour can drive the air before it, so that the major part of the condensing space is filled with the pure vapour.

The Liebig condenser is obviously far from fulfilling these conditions; the worm condenser has nearly all the bad points of the Liebig, of which it is really only a coiled form. The spherical condensers also, now becoming so popular on account of their smallness, are as a rule made with the condensing chamber much too large.

The general plan of the form now exhibited is shown in the accompanying sketches. The vapour enters through the tube A into the condensing space B between the two tubes, C and D; after condensation the distillate passes out through E. The cooling water passes down the tube F into the interior of C, which it fills, and, running over the rim at the top, passes in a thin stream down the outside of D, finally flowing away through the escape pipe G. When used as a reflux condenser, the vapour, of course, passes up through E. After allowing the liquid to get into full ebullition, so that the air is driven out of the condensing space, the mouth of A may be completely closed with a cork, thus avoiding even the possibility of loss with volatile liquids such as ether. The apparatus must obviously always be used in one and the same position, *i.e.*, with its axis vertical. This is a distinct advantage, because it is very easily so supported, and because, when thus disposed, it takes up a minimum of space on the bench. An absolutely vertical position, however, is not necessary. It is preferable that the top of the condenser should slope, if at all, towards the flask or retort from which the liquid is being distilled. To obtain the greatest possible efficiency in working, special steps should be taken to ensure the even distribution of the water over the outside of the condenser, as the adhesion between the water and the glass or metal, and the surface tension of the water itself, are reduced to a minimum at high temperatures, so that the cooling water tends to run in narrow streams outside D, instead of spreading out. This is easily remedied by covering D externally, either partially or entirely, with some absorbent material, such as blotting-paper or linen (the latter as a permanence is to be preferred). In addition to this a cork I (Fig. 1), with a number of vertical grooves cut round its outside, is inserted into the mouth of C. The cork is perforated for the cold-water supply tube F, and the water leaves the interior of C by the grooves in the cork, thus getting well distributed round the outside of the condenser. This little device has

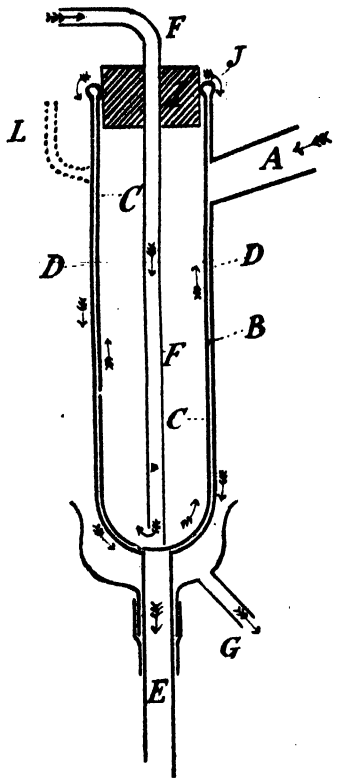


FIG. 1.

the further advantage of preventing any water getting spilt if the condenser receives a sudden knock or jerk whilst in action. With metal condensers the cork may be replaced by a metal disc H (Fig. 2) attached to the cold-water supply tube, and arranged so as to leave space either below it (as in the sketch), or outside it for the water to flow out of the cup on to the exterior of D.

To ensure the utilization of the whole available cooling surface, a wire or band of metal K (Fig. 2) may be coiled spirally round the interior of the condensing space, thus converting it into a flat spiral channel.*

Efficiency.—This may be treated both from a theoretical and from a practical standpoint.

Theoretically it is obvious that the new form has many points of superiority over the Liebig, the worm, or even the spherical condenser as at present made.

The condensing space in the small metal condenser exhibited, of which the outside dimensions of the body are $\frac{15}{16}$ inch × $4\frac{1}{4}$ inch, is only 9 cubic centimetres. The glass one shown — measuring outside $1\frac{3}{8} \times 5\frac{3}{8}$ inches—has an internal capacity of 30 c.c., while a Liebig's condenser, of which the water-cooled part measured $19 \times \frac{7}{16}$, was found to hold 55 c.c., and a ball condenser $3\frac{1}{2}$ inches in diameter was found to contain over a hundred cubic centimetres.

That the practical performance of the condenser is at least equal to its theoretical promise may be seen from the following figures :—

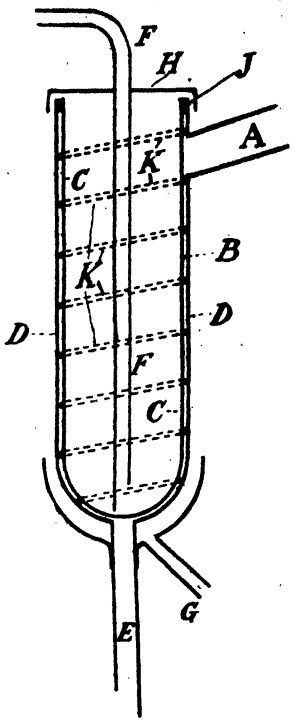


FIG. 2.

	Small Metal Condenser. (1).	(2).	(3).	Glass Condenser. (4).	(5).
Temperature of effluent cooling water ...	52° C.	65° C.	76° C.	61° C.	47·5° C.
Temperature of distillate	48° C.	60° C.	61° C.	41° C.	35·5° C.
C.c. of cooling water used per hour ...	40,000	40,000	7,200	6020	2093
C.c. of distillate per hour	2,642	4,128	1,017	765	355
Ratio of quantity of distillate to that of cooling water	$\frac{1}{15·14}$	$\frac{1}{9·69}$	$\frac{1}{7·8}$	$\frac{1}{7·87}$	$\frac{1}{5·9}$

The resistance of the metal condenser, working as in column (2), was roughly equal to one inch of mercury, the volume of the steam condensed in one second being two hundred times the actual capacity of the condenser. When the water supply was stopped, the resistance rose to $1\frac{3}{4}$ inches of mercury.

One of the metal condensers with a body measuring $4\frac{1}{4} \times 1\frac{7}{8}$ outside, but with an unnecessarily large condensing space, when connected up to a high-pressure boiler

* For all ordinary purposes this is quite unnecessary.

(working at 43 lbs. pressure), yielded 8,040 c.c. of distillate per hour. It was, however, working under somewhat disadvantageous circumstances, as it was found impossible to fix it except in a somewhat slanting position, so that the water ran mainly down one side of the exterior.

The glass condensers have not yet been tested as to their extreme capacity, but they have been found ample for any and all of the ordinary laboratory operations, in which 500 c.c. per hour may be taken as the maximum rate of distillation. The metal forms can be used with a ratio of distillate to cooling water as high as $\frac{1}{3}$ without any steam escaping, though the distillate itself is nearly boiling.

In addition to its great efficiency and extremely small size and weight, the new form of condenser has the following advantages :—

It is always used in one and the same position, *i.e.*, *vertical*, in which it takes up a minimum of bench room, and in which also it is very easily fixed, a little bracket projecting from the wall, or a hole in a block sliding up and down a retort stand being ample for the purpose. If greater rigidity is required, the upper end may also be held by a wire or spring clamp attached to the top of the tube F. It can be made, if necessary, in both metal and glass, so that the inner tube C can be withdrawn from the outer. The whole of the interior of the condensing space is thus at once open to view, and can be wiped dry and clean with a duster.

To effect this in the metal condensers a slip-in or a screw joint is made at J (Fig. 2); in glass condensers the fused joint at J (Fig. 1) is replaced by an india-rubber joint. Owing to the cooling water running down *outside* the condenser, it is practically impossible for the escape tube to get choked up with fur, as happens sometimes to a Liebig's condenser after long-continued use. The escape tube G should be made about twice the diameter of the cold-water delivery tube as a further safeguard.

Owing to the vertical position and to the small diameter of the extremity, the condenser is very easily connected in an air-tight manner to a receiver, so that, with the addition of a little side-tube at L for attachment to a pump, distillation may be conducted under reduced pressures. Or, if a suitable mercury valve be attached to L, and a little air be sucked out at the commencement of the operation, distillation or digestion may be carried on in an entirely closed space.

In tropical climates, or with liquids of exceptionally low boiling-point, the tube C may be filled with ice, and the water allowed to flow up through it.

The very thin layer into which the water is spread when pouring down the outside of the condenser greatly facilitates evaporation, so that the latent heat of vaporization, which has mainly to be derived from the vapour undergoing condensation, effects a great saving in the cooling water used. This is especially the case with liquids boiling above 100° C., but may be easily shown in the case of water by cutting down the supply of cooling water to a minimum, when the water at the top of C is sometimes as much as 10° hotter than that issuing from G.

Finally, its simplicity renders it easy of construction in almost any material likely to be employed.

The condensers can be obtained from Messrs. J. J. Griffin and Sons, Garrick Street, and from Messrs. C. E. Müller and Co., High Holborn.

THE ANALYSIS OF MARMALADE.

By L. K. Boselby, Chemist to Messrs. T. Keiller and Son, Limited.

(*Read at the Meeting, March* 16, 1898.)

Having had occasion lately to work out some methods for the analysis of marmalade and jams, I have thought that a description of these, together with several results showing the composition of marmalade, might be of interest to the members of this Society.

Very few analyses on this subject have been published up to the present time—indeed, a few made by Mr. Carter Bell, the Analyst for Chester, are the only ones I know of. These analyses, which have been published quite recently, are not at all in agreement with any which I have myself made; among them are four analyses of marmalade, of which I give particulars, together with a few quotations from the report: "In the manufacture of jam on a large scale glucose, or inverted sugar, is often used in the place of cane. But to attempt to estimate this adulteration would be hopeless, for in the manufacture of jam the acids of the fruit convert the cane-sugar, more or less, into glucose."

The following analyses are then given:

	Water.	Glucose.	Cane-sugar.	Ash.
1. Marmalade ...	20·5	21·05	37·77	0·305
2. ,, ...	21·8	15·62	34·38	0·31
3. ,, ...	22·5	24·39	25·61	0·30
4. ,, ...	19·5	21·73	14·63	0·26

From the remarks quoted above, it would seem that Mr. Carter Bell is unable to distinguish between the product of the action of sulphuric acid on starch, usually known as glucose, and the product of the action of a fruit acid on cane or beet sugar, usually known as invert sugar.

The sugar estimated in these analyses appears to me to be very much too low; for if the last sample be taken, and the water, glucose, and cane-sugar added together, they amount to 55·86 per cent., a figure which leaves 44·14 per cent. of dry fruit (the water being estimated), which is not possible. My own experience goes to prove that marmalades from various sources contain from 2½ to 5½ per cent. of dried fruit. The probability is that both the figures for water and for sugar are much too low, as I never met with a marmalade with so low a water as 19·5 per cent., and one which contained 36·36 per cent. of total sugar, more than half of which was glucose, would be little thicker than water.

The following are the methods in use in my laboratory:

Water.—This is estimated by taking a flat-bottomed porcelain basin containing a glass rod, and weighing into it about 7 to 8 grammes of the well-mixed marmalade. This is warmed, and dissolved in a few c.c. of 40 per cent. alcohol. From 12 to 15 grammes of silver-sand, which has been previously dried, if necessary, are now weighed into the basin, and the marmalade, sand, and alcohol thoroughly mixed with the glass rod. The basin is then heated on the water-bath for an hour, when 5 c.c. of absolute alcohol are added, and the basin again heated for one hour; after this it is

transferred to the air-bath and dried at 95–100° C. for thirty-six hours, or until the weight is nearly constant.

Acidity.—This is estimated by weighing out 20 grammes of the marmalade, titrating with $\frac{N}{10}$ soda, with phenolphthalein, or litmus-paper as indicator. The number of cubic centimetres used, multiplied by 0·035, gives the percentage calculated as citric acid.

Sugars.—The following methods are the best, supposing only cane and invert sugar to be present: Weigh out 65·12 grammes of the well-mixed marmalade in a small beaker, add successive quantities of *cold* water—say, 50 c.c.—stir well, and decant into a 250 c.c. flask. Then transfer the peel to the flask, add basic lead acetate, until the solution is only slightly acid, make up to 250 c.c., and mix well. It is best not to add sufficient basic lead acetate to neutralize the solution, as one is apt to get lead in the filtrate, this being an objectionable feature, as a slight precipitate is produced on adding acid to invert the solution which is apt to interfere with the polarimeter reading. The contents of the 250 c.c. flask are now filtered through a dry filter, and polarized at $t°$ C. Fifty c.c. of the filtrate are placed in a flask, and 5 c.c. of pure, strong HCl added, and the flask is heated on the water-bath till a thermometer suspended in the middle of the flask indicates 68° C. in ten minutes. Cool quickly to $t°$, and again polarize. Then,

$$\text{Cane-sugar} = \frac{\text{Direct} - \text{inverted reading}}{144 - \frac{t°}{2}} \text{ (this is the well-known Clerget formula).}$$

$$\text{Invert sugar} = \frac{(\text{Cane-sugar} - \text{direct reading})\ 100}{44 - \frac{t°}{2}}.$$

If glucose be present it will be indicated by the inverted reading being + instead of −, or, at all events, being very much smaller than usual. If this be the case, it will be necessary also to determine the cupric reducing power (in duplicate).

The details of the method are as follows: Weigh out 13·024 grammes of pure cane sugar, make up to 100 c.c., add 10 c.c. strong HCl, and invert as before. Cool, take 11 c.c. of this solution, neutralize, and make up to 100 c.c. This I call solution *a*.

Dilute 20 c.c. of the filtrate used for direct polarization, and make up to 100 c.c. This I call solution *b*.

Take two beakers, and in each place 25 c.c. of Soxhlet's copper solution and 25 c.c. of Soxhlet's alkaline tartaric solution and 40 c.c. of water, add to one 10 c.c. of solution *a*, and to the other 10 c.c. of solution *b*. Place both over flames and bring to the boil in four minutes, and boil for four minutes longer, filter off the reduced cuprous oxide, and weigh either as metallic copper or copper oxide. The cupric reducing power of the marmalade, calculated as percentages of invert sugar, will be found by the following formula:

$$= \frac{\text{Cu (or CuO) from } b}{4 \times \text{Cu (or CuO) from } a} \times 100.$$

The reason for making solution *b* four times as strong as solution *a* is that the

cupric reducing power of a marmalade is somewhere in the neighbourhood of 25 per cent.

Calculation of the Percentage of Glucose.—I need hardly point out that "glucose" is not a chemical entity, but is a mixture of maltose, dextrin, dextrose, and probably intermediate compounds. The following table will give the composition of commercial glucoses. The first nine are taken from a paper by Weber and McPhearson ("Proceedings of the Eleventh Annual Convention of the Association of Official Agricultural Chemists," p. 126):

	1.	2.	3.	4.	5.	6.	7.	8.	9.	10.
Total solids	78·9	80·1	80·8	80·3	85·6	87·4	80·0	81·1	86·6	78·9
$[\alpha]_D$ of solids	133·3	132·9	120·6	135·4	132·1	127·8	149·3	130·3	134·9	143·1
Cupric reducing power of solids ...	56·6	57·0	60·6	54·9	56·7	55·9	43·6	50·6	48·8	48·5

Analysis No. 10 is by H. D. Richmond. The average of these ten samples, which may be taken to fairly represent the mean composition of commercial glucose, is: Total solids, 81·9; $[\alpha]_D$ of solids, 133·98; cupric reducing power of solids, 53·31.

From the paper referred to, I find that Weber and MacPhearson found that commercial glucose, on the average, gave, in a solution containing 13·024 grammes per 100 c.c., a

Direct reading	...	...	85·45 sugar degrees.
Inverted reading	...	...	84·9 ,,

The inverted reading is done by the method described above, which must be strictly adhered to, Herzfeld's modification having given a much larger difference. It is seen that the change on inversion is practically nil, and therefore cane-sugar can be estimated in the presence of commercial glucose with very fair accuracy. The average $[\alpha]_D$ of the solids of commercial glucose (133·98) is practically twice that of cane-sugar, and it may be assumed without much error that 1 per cent. of glucose solids will polarize 2 per cent. of cane-sugar. When cane-sugar is inverted, 100 parts become 105·3:

$$C_{12}H_{22}O_{11} + OH_2 = C_6H_{12}O_6 + C_6H_{12}O_6.$$

The cupric reducing power of inverted cane-sugar is therefore 105·3. The cupric reducing power of glucose solids, 53·13, is practically half this, and it may be assumed without much error that 1 per cent. of glucose will have a cupric reducing power equal to 0·5 per cent. of cane-sugar.

To calculate the percentage of cane-sugar, invert sugar, and glucose in a marmalade, the following formulæ can be derived from the facts above mentioned:

$$\text{Cane-sugar} = \frac{\text{Direct} - \text{inverted reading}}{144 - \frac{t^\circ}{2}}.$$

$$\text{Inverted sugar} = \frac{\text{Cane-sugar} - \text{direct reading} + 4 \text{ cupric reducing power}}{4 + 44 - \frac{t^\circ}{2}}$$

$$\text{Glucose} = 2(\text{cupric reducing power} - \text{inverted sugar}).$$

It must be borne in mind that the inverted sugar is given by all the above formulæ as percentages of cane-sugar inverted, and not as percentages of actual invert sugar present. This is done for the sake of convenience, as by adding the cane and inverted sugar together the percentage of cane-sugar present will be given.

As a test analysis, a marmalade, of which the composition was unknown to me, was made up containing:

Cane-sugar	40·0 per cent.
Glucose	20·0 ,,

The figures found on analysis were as follows:

Direct polarization	+61·2 at 16·5 C.
After inversion	+18·2 ,,
Cupric reducing power	16·48

From these results the composition was calculated as follows by the above formulæ:

Cane-sugar ... 31·7 } 40·0 total
Inverted sugar ... 8·3 }
Glucose ... 16·3 = glucose containing 81·9 per cent solids = 19·9 per cent.

This result seems to indicate that cane-sugar, invert sugar, and commercial glucose can be estimated with sufficient accuracy in the presence of each other.

The following analyses give the composition of nearly all the best known brands of marmalade on the market, the results having been all obtained by the methods given above:

	1.	2.	3.	4.	5.
Moisture ...	33·3	33·1	27·7	30·6	31·5
Sugar	37·2	42·1	25·6	32·6	33·2
Inverted sugar ...	25·0	21·0	41·8	32·7	30·0
Acidity	0·5	0·3	0·4	0·4	0·5
Undetermined ...	4·0	3·5	4·5	3·7	4·8
	100·0	100·0	100·0	100·0	100·0

	6.	7.	8.	9.	10.
Water	31·3	26·6	30·0	25·3	31·7
Sugar	34·7	35·6	33·9	22·8	32·8
Inverted sugar ...	23·9	30·4	11·1	38·2	30·4
Glucose ...	8·7	5·0	22·9	11·6	none
Acidity	0·4	0·5	0·4	0·4	0·5
Undetermined ...	1·0	0·9	1·7	1·7	4·6
	100·0	100·0	100·0	100·0	100·0

	11.	12.	13.	14.	15.
Water	30·7	31·1	40·6	28·6	27·7
Sugar	19·7	32·9	31·4	44·7	44·0
Inverted sugar ...	34·1	17·9	21·5	21·9	22·9
Glucose ...	13·3	14·3	none	none	none
Acidity	0·6	0·4	0·5	0·6	0·5
Undetermined ...	1·6	3·4	6 0	4·2	4·9
	100·0	100·0	100·0	100·0	100·0

	16.
Water	28·6
Sugar	24·3
Inverted sugar	42·8
Acidity	0·6
Undetermined	3·7
	100·0

The sample labelled No. 8 contained added gelatin or isinglass, and the sample No. 16 was one of Messrs. Keiller's marmalade, which had been kept for six years; hence the abnormally high invert sugar. Nos. 11 and 12 were preserved with salicylic acid. The amount of invert sugar formed in a marmalade seems to be due to three causes: Firstly, the amount of acid present; secondly, the length of time the cooking is continued; and, thirdly, the length of time it has been kept. The figures for undetermined matter vary somewhat, but as the error of separating and estimating a complicated mixture of sugars is thrown on this figure it is not surprising.

Sugar is naturally present in oranges, and a correction has been applied for the amount of fruit-sugar present in a marmalade; it consists in subtracting 0·1 sugar degrees from the + reading, and 0·3 from the – reading—that is, supposing the normal quantity of marmalade to have been weighed out, namely, 65·12 grammes per 250 c.c.

In conclusion, I should like to express my thanks to Mr. H. Droop Richmond for help in confirming some of the results given in this paper.

Discussion.

The President having invited discussion,

Mr. A. C. Chapman inquired what assumption Mr. Boseley made in regard to the composition of glucose in the method he had adopted. Ordinary commercial glucose (a mixture of dextrose, maltose, and occasionally dextrin) varied very much in composition. The proportion of maltose, for instance, might be as little as 4 or 5 per cent., or as much as 25 per cent. Some samples contained no dextrin at all, while others contained a fairly considerable quantity. It would be necessary to assume a constant composition as a basis for purposes of analysis and calculation.

Mr. Hehner observed that an important reference had been made in the paper to salicylic acid. Salicylic acid was often found in jam, and had given rise to a good deal of discussion. Mr. Boseley now stated that it was added to jam solely as a means of adulteration, and in view of this he (Mr. Hehner) thought that its presence in jam might be somewhat more vigorously condemned than hitherto. The excuse that it prevented the preserve from going bad, and that it was not particularly harmful, might justify its being passed; but when it was distinctly understood that it also afforded a means of adding water, the matter became very serious.

Mr. L. K. Boseley said, in reply to Mr. Chapman's question, that the basis the calculations had been worked out upon was the cupric reducing power of the glucose, and also the $[\alpha]_D$ of the glucose solids. He was sorry not to have had time enough to make this clear when reading the paper, but Mr. Chapman would understand that the amount of dextrin, dextrose, etc., which the glucose contained did not enter into the calculation, that being based on the average cupric reducing power, etc., of glucose.

NOTE ON THE VOLUME CONCENTRATION OF CONDENSED MILK.

By A. McGill.

The purchaser of condensed milk, as a rule, thinks of its concentration in terms of volume rather than weight; and values a pint of the article more or less highly as he takes it to represent more or less than, say, three pints of the milk used in its preparation. A. H. Allen (see Analyst, xxi., p. 281) has given a formula for calculating, from the results of analysis, the volume concentration of any sample. His formula involves a knowledge of the specific gravity of the sample, and assumes the normal character of the milk from which the condensed article is made.

I would suggest the *solids-not-fat* instead of the *total solids* as a basis for calculation of the concentration. It is mainly in the partial or entire removal of the fat before manufacture that condensed milks differ from each other; and although, in the case of a whole milk, it makes little difference whether total solids or solids-not-fat is employed in the calculation, the error is very considerable when a skimmed milk is in question.

The following examples, taken from the analyses of Pearmain and Moor (Analyst, xx., 272), illustrate this:

Brand.	Concentration based on Total Solids = 12·5 per cent.	Concentration based on Solids-not-Fat = 8·5 per cent.
Milkmaid	... 3·85 ...	... 4·02
Lancer	... 3·26 ...	... 4·74

These figures are calculated by Mr. Allen's formula, in which the density of normal milk is taken as 1, and that of condensed milk as 1·28. My own experience shows that most samples of sweetened condensed milk have a gravity of 1·31 to 1·33; and I prefer to use the number 1·03, instead of unity, as the average density of normal milk. The density of condensed milk is not easily taken upon the sample itself. I prepare a solution of 50 grammes to 250 c.c., and from the density of this solution calculate that of the original sample by the formula:

$$\text{Original density} = \frac{1}{6 - 5a}, \quad \text{when } (a) = \text{density of the dilute milk.}$$

If these changes be made, the numbers already given for Milkmaid and Lancer brands will become 4·01 and 4·75, instead of 4·02 and 4·74; so that, for practical purposes, it is of little consequence which pair of numbers is used. If, however, the true density of the sample be used in calculation, while the density of normal milk is taken as unity, the numbers become 4·13 and 4·84 respectively.

The formulæ used may be written as follows:

Let n = solids-not-fat in normal milk.
" n_1 = solids-not-fat in samples.
" d = density normal milk.
" d_1 = density of sample.
" f = fat in milk used in manufacture.
" f_1 = fat found in sample.
" c = volume concentration.

$$\text{Then, } c = \frac{n_1 \times d_1}{n \times d} = \frac{n_1 \times d_1}{8{\cdot}5 \times 1{\cdot}03} = \frac{n_1 \times d_1}{8{\cdot}755}.$$

$$f = \frac{n \times f_1}{n_1} = \frac{8{\cdot}5\, f_1}{n_1}.$$

Thus, the fat percentage in the original milk used in preparing the Milkmaid brand is found to be 3·515.

LABORATORY OF THE INLAND REVENUE DEPARTMENT, OTTAWA,
January 15, 1898.

THE INSTITUTE OF CHEMISTRY.

THE annual dinne of the Fellows and Associates of the Institute of Chemistry took place last night at the Trocadero Restaurant, Piccadilly Circus. The president, Dr. T. Stevenson (official analyst to the Home Office), occupied the chair, and the large company present included Lord Reay, Mr. Justice Byrne, Sir J. Evans (treasurer Royal Society), Sir E. Frankland, Sir H. T. Wood (secretary of the Society of Arts), Mr. K. E. Digby, Dr. Bernard Dyer, Dr. W. J. Russell, Dr. Thorpe, Mr. T. H. Elliott, Mr. J. F. Moulton, Q.C., Mr. H. Kearley, M.P., Mr. H. H. Cozens-Hardy, M.P., Professor Dewar, Dr. J. H. Gladstone, Dr. Ludwig Mond, Mr. W. Hills (president of the Pharmaceutical Society), Dr. H. E. Armstrong, Dr. W. J. Sykes, Dr. J. A. Voelcker, Mr. Otto Hehner, Professor J. M. Thomson, Mr. D. Howard, Mr. R. J. Friswell, Mr. T. Fairley, Dr. Corfield, Dr. Wynter Blyth, and Mr. R. B. Pilcher (secretary). Mr. Justice Byrne proposed "The Institute of Chemistry," and the President, in responding, mentioned that the register of the society now contained the names of 826 Fellows and 120 Associates, while there were over 180 registered students training for the examination at the various colleges recognised by the Institute. He looked forward to the time when professional chemists would be endowed with the power of conferring diplomas, and exercising the same restrictive functions as were already possessed by the professions of the law and physic. Other toasts followed. (Abridged from the *Times*.)

ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

FOODS AND DRUGS ANALYSIS.

Black Pepper from Mangalore. T. F. Hanansek. (*Zeit. für Untersuch. der Nahr. und Genussmittel*, 1898, 153.)—Among a collection of samples of pepper supplied by Messrs. Lewis and Peat, of London, was a variety of black pepper from Mangalore, which differs considerably from the sorts usually met with. The author is not aware if it is on the English market to any large extent, but in Germany and Austria it is quite unknown. Its place of origin, Mangalore, is a town on the west coast of India, between Goa and Cochin, and therefore in the heart of the pepper-growing district. The pepper-corns are about 7 mm. in diameter, of a deep black colour, and either nearly spherical or somewhat egg-shaped. One hundred of the

peppercorns weigh 8·6 grammes. They possess a very powerful pepper taste and odour. The ash amounts to 3·43 per cent. The microscopic examination of the husk reveals much the same general structure as the husk of ordinary pepper. Illustrations are given showing the microscopic appearance of sections of the peppercorns, the cells, etc. H. H. B. S.

Glucose in Butter. C. A. Crampton. (*Jour. Amer. Chem. Soc.*, 1898, xx., 201-206.)—The author states that the method found most satisfactory by the manufacturers for preserving butter for export to tropical countries is by the addition of considerable quantities of salt and of glucose. The following table gives the results of the analyses of three samples of such prepared butter:

	No. 2,434. Butter exported from the United States to Brazil.	No. 2,443. Butter exported to Brazil and returned to U.S.A.	No. 2,460. *Beurre Rouge* exported to Guadeloupe.
Water	16·29	18·93	21·60
Casein (N × 6·25) ...	1·19	1·06	0·81
Ash	7·00	6·19	16·42
Fat	72·16	69·67	51 15
Glucose (by difference) ...	3·36	4·15	10·02
	100·00	100·00	100·00

The characteristics of the fat separated from these three butters were not abnormal, though the Reichert-Meissl value of No. 2,443 was somewhat low (25·4). For this reason the Brazilian authorities rejected the butter, but the glucose escaped their notice. This butter when returned was still passable.

A sample of the glucose used for this purpose was found by the author to consist of the ordinary syrup known as confectioner's glucose. It contained 16·5 per cent. of water, and gave $[\alpha]_D = 119·3$.

As to its detection in butter, it is shown that on extracting the fat with ether, and determining the casein by loss on ignition, the results are abnormally high and widely different to those obtained by calculating the casein from the amount of nitrogen; whereas with pure butter the figures are in close agreement.

For a direct determination of the glucose, 10 grammes of the butter are treated with successive portions of hot water in a separatory funnel, and the resulting solution made up to 250 c.c. A slight reduction of Fehling's solution by this solution might be due to milk-sugar or some of the albuminoid substances, but any considerable reduction must be attributed to the presence of sugar or glucose. The optical rotation can be determined in the same solution previously clarified by means of alumina cream or acid mercuric nitrate solution.

The figures given by the author show that either method gives reliable results.

C. A. M.

ORGANIC ANALYSIS.

A Colour Reaction of Ethylic Aldehyde. L. Simon. (*Comptes rendus*, vol. cxxv., p. 1105; through *Rev. Chim. Analyt. appl.*, vol. vi. [5], p. 79.)—By adding to a dilute aqueous solution of ethylic aldehyde a few drops of trimethylamine followed by several drops of an almost colorless solution of sodium nitro-prusside, there gradually develops a beautiful blue coloration, which is intense in the case of a 1 per 1,000 solution, and apparent down to a degree of dilution equal to $\frac{1}{25000}$. No other aldehyde or ketone substance gives this reaction, neither does pure ether, although the coloration appears when the latter contains $\frac{1}{2000}$ of aldehyde. Ammonia cannot replace trimethylamine, since it prevents the appearance of the coloration, and destroys it if already formed. C. S.

Colour Reaction of Phenylhydrazine. L. Simon. (*Comptes rendus*, vol. cxxvi., p. 483; through *Rev. Chim. Analyt. appl.*, vol. vi. [6], pp. 79, 80.)—When phenylhydrazine solution is heated for a few moments with a few drops of an aqueous solution of trimethylamine followed by a few drops of sodium nitro-prusside, and by concentrated potash, a bright blue coloration is produced, which is greenish in presence of an excess of nitro-prusside, and deepens on adding the potash, and is converted into sky-blue if acetic acid be added as well. The coloration, which enables $\frac{1}{50000}$ part of phenylhydrazine to be detected, is distinguishable from that produced by ethyl aldehyde (see preceding abstract) by its persistence in presence of potash, ammonia, and acetic acid; it is, however, fugitive, and disappears in about a quarter of an hour. Whilst alcohol, ether and ammonia do not prevent the reaction, chloroform and benzene retard it, as do also mineral and organic acids previous to the addition of potash. It appears to be exclusively characteristic for phenylhydrazine and the derivatives of the latter substituted in the aromatic nucleus. C. S.

Characteristic Colour Reaction of Cotton-seed-Oil. G. Halphen. (*Ann. Chim. Analyt.*, vol. iii. [1], pp. 9-11.)—The development of the red coloration produced by cotton seed-oil in presence of carbon disulphide can be accelerated, so as to be utilizable for the purposes of qualitative analysis, by the presence of free sulphur in the solvent, and the addition of amyl alcohol.

Equal volumes (1 to 3 c.c.) of the oil under examination, amyl alcohol, and carbon disulphide containing 1 per cent. of dissolved sulphur, are placed in a test-tube, which is then partly immersed in a boiling brine-bath for ten to fifteen minutes. A red or orange coloration denotes the presence of cotton-seed-oil. If, however, no change is observed, another 1 c.c. of the same carbon disulphide is added, and heat again applied for five to ten minutes, this operation being repeated in the event of the result again proving negative. As little as 5 per cent. of cotton-seed-oil can be readily detected in this manner. C. S.

Analysis of Fats. Melting-points of Cholesterin and Phytosterin. A. Bömer. (*Zeit. für Untersuch. der Nahr. und Genussmittel*, 1898, 81.)—The melting-point of cholesterin from animal sources (excluding butter) fluctuates, according to most observers, between 144·5° and 146° C., and of phytosterin from vegetable sources between 131·5° and 138° C. The author has made a large number of determinations of the melting-points of cholesterin and phytosterin extracted from different animal and vegetable sources by the method recently published by him (*Zeit. für Untersuch. der Nahr. und Genussmittel*, 1898, 21-49; ANALYST, xxiii., 42), and in order to arrive at as exact results as possible, he in every case repeatedly recrystallized the products, determining the melting-points after each crystallization, until constant. The determinations of the melting-points of thirty-eight specimens of cholesterin from various kinds of pig-fat gave results as follows:

	First Indications of Melting.	First Indications of Flowing.
Minimum	146·0°	146·5°
Maximum	148·0°	148·3°
Mean	146·7°	147·4°

According to Salkowski, the melting-point of cholesterin from butter is abnormal. He found that, even after repeated crystallization, the product, though apparently quite pure and white, melted persistently at about 5° below the ordinary melting-point of cholesterin, a peculiarity which he explained by supposing that it contained a proportion of phytosterin. The author's experiments, however, show that, if carefully purified, butter cholesterin melts quite normally at 146° and over. Crude cholesterin prepared from butter is by no means so pure as that prepared from lard. It contains two impurities in considerable proportions—viz., coloring matter and lecithin, and while the latter is very difficult to separate, its presence has a marked effect in reducing the melting-point of the product. The author further observes that if phytosterin were present in conjunction with cholesterin, its presence would certainly be revealed by the appearance of the crystals.

The determinations of the melting-points of specimens of phytosterin prepared from various vegetable fats gave results as follows:

	First Indications of Melting.	First Indications of Flowing.
Minimum	135·5°	136·0°
Maximum	141·0°	141·5°
Mean	137·5°	137·8°

Experiments upon the melting-points of mixtures of cholesterin and phytosterin showed that the melting-point of a mixture approximates to that obtained by calculation from the melting-points of its components, though a mixture of 3 parts of phytosterin and 1 of cholesterin gives a slightly lower, and a mixture in the reverse proportions a slightly higher, melting-point than the calculated. Mixtures of cholesterin and phytosterin cannot be separated into their components by crystallization.

The determination of cholesterin may be found useful for the following purposes:

1. The detection of vegetable oils in cod-liver-oil.
2. The detection of cotton-seed-oil in lard, etc.

3. The detection of margarine in butter, in cases where the Reichert-Meissl method fails. Margarine being rarely free from vegetable oils, the determination of phytosterin affords a valuable means for its detection. H. H. B. S.

Technical Analysis of Bone-fat. A. A. Shukoff and P. J. Schestakoff. (*Chem. Rev. Fett u. Harz-Ind.*, vol. v. [1], pp. 5-8; [2], pp. 21-23.)—1. *Estimation of Water.*—The ordinary method of determination, by drying 5 grammes of fat at 100° to 110° C. in the oven until of constant weight, is adopted. The time occupied depends on the purity of the fat; bone fats containing lime salts often require over twenty-four hours owing to the tenacity with which these impurities retain moisture.

The Tate method of heating at 130° C. until the evolution of gas ceases gives results below the truth. When lime salts are present, the use of a current of carbon dioxide for accelerating drying is not feasible, owing to the frothing produced.

2. *Extraneous Admixtures.*—These comprise (organic) dirt, sand, calcium phosphate, and lime soaps. In the ordinary method of estimation by dissolving the fat in petroleum spirit or carbon disulphide, considerable differences exist in the amount of insoluble residue, the influential factors being the solvent, the temperature, the duration of the operation, and the presence or absence of water. Carbon disulphide dissolves more of the lime salts than does petroleum spirit, and in most cases reprecipitation occurs on standing or cooling. Undried fat leaves a much larger proportion of lime salts as insoluble residue than dried fat.

Apart from the question whether, in view of their high percentage of fatty acid, lime soaps can properly be regarded as impurities (though their presence is inconvenient when the fat is to be converted into soap), the solution method does not give accurate results, and other means must be employed (see §§ 3 and 4).

3. *Ash.*—This must be determined by combustion of the entire fat, and not merely from the residue left by solvents. The principal constituent being calcium carbonate and oxide, the amount of calcium can be determined by titration, and the amount of lime soaps by calculation from the result, 260 being taken as the average molecular weight of the fatty acids. When sand, calcium phosphate, etc., are present, a quantitative analysis of the ash must be made, but this is seldom necessary.

4. *Direct Estimation of the Pure Fat.*—In place of the usual, but erroneous, method of determining this chief constituent by difference, the authors propose the following:

About 10 grammes of fat are gently heated, with frequent shaking, for about an hour on the water-bath, with 3 to 5 drops of strong hydrochloric acid to decompose the lime soaps, and are then dissolved in 40 c.c. of light petroleum spirit. The solution is decanted from the acid through a tared filter, and is then distilled along with the (two or three) washings to drive off the bulk of the solvent, the pure fat thus obtained being finally dried at 100° to 110° C., with the acid of a current of carbon dioxide, until constant.

The residual acid and dirt are rinsed with water through the same filter, and the impurities determined therefrom.

Water can be estimated by difference to check the results obtained by direct determination.

5. *Unsaponifiable Matter.*—The results furnished by the Allen and Thomson method are higher than by that of Morawski and Demsky, owing to the more complete extraction of the cholesterin. The former method, however, is inaccurate owing to the solution of a portion of the soap in the ether employed, and the authors therefore recommend the following modification :

Five grammes of fat and 25 c.c. of 8 per cent. alcoholic caustic soda are evaporated to dryness, taken up with 80 c.c. of water and extracted with an equal volume of ordinary ether in a separating funnel, the separation of the liquids being facilitated, if necessary, by the addition of alcohol, but avoiding an excess of the latter. After the third extraction the ethereal solution is evaporated without washing, the residue is rendered alkaline by normal caustic soda and re-dissolved in petroleum spirit, which solution yields on filtration and evaporation an ashless residue.

The technical importance of the unsaponifiable residue depends on the use to which the fat is put; for soap-making it must be looked on as an impurity and deducted from the total fat, whilst for the stearin industry the presence of cholesterin is no drawback.

The solidifying-point of the fatty acids is determined by the original Dalican method. C. S.

Precipitation of the Albumoses by Zinc Sulphate. K. Baumann and A. Bömer. (*Zeit. für Untersuch. der Nahr. und Genussmittel*, 1898, 106.)—In a former paper by one of the authors (A. Bömer, *Zeit. anal. Chemie.*, 1895, 562) zinc sulphate was proposed in place of ammonium sulphate as a precipitant for the albumoses The authors have further investigated the subject, and arrived at the following results :

1. If 1 c.c. of dilute sulphuric acid (1 : 4) be added to 50 c.c. of solution, the albumoses are precipitated as well with zinc sulphate as with ammonium sulphate.

2. Ammonium salts, tyrosine and kreatine are not precipitated by zinc sulphate. Leucine is thrown down in small quantities, but considering the small amount present in meat preparations the error is not material. Leucine and tyrosine are, on the other hand, both separated in considerable quantities by ammonium sulphate.

3. The meat bases are as completely separated by phosphomolybdic acid in the filtrate from the zinc sulphate precipitation as they are in an aqueous solution, and the peptones are even more completely separated.

4. The filtrate from the precipitation of the albumoses can be precipitated directly with phosphomolybdic acid, thus avoiding the error caused by the different nitrogen contents of the albumin and gelatin albumoses.

5. Ammonia and kreatine are separated from solutions by sodium phosphomolybdate with almost quantitative exactness.

The authors propose the following method for the determination of the albumoses and peptones in meat extracts and peptone preparations : Fifty c.c. of solution

containing about 1 gramme of dry substance are freed from insoluble and coagulable albumin, and mixed with 1 c.c. of dilute sulphuric acid (1 : 4) and sufficient zinc sulphate added to supersaturation. The precipitate is filtered off and washed with a slightly acidified saturated solution of zinc sulphate, and the nitrogen determined by Kjeldahl's method. In the filtrate the peptones, meat bases, and ammonia are precipitated by sodium phosphomolybdate. The precipitate is filtered off, and the nitrogen also of this determined by Kjeldahl's method. The ammoniacal nitrogen is determined, either in the aqueous solution, or, better still, in a second phosphomolybdate precipitate by distillation with magnesia. The amount so found being deducted from the total nitrogen in the phosphomolybdate precipitate gives by difference the nitrogen present as peptones and meat bases with, under certain circumstances, small quantities of leucine. H. H. B. S.

INORGANIC ANALYSIS.

Volumetric Determination of Antimony. H. Causse. (*Comptes rendus*, vol. cxxv., p. 1100; through *Rev. Chim. Analyt. appl.*, vol. vi. [3], p. 31.)—The method is based on the conversion of antimonious acid into antimonic acid by iodic acid. As reagents the author employs: (1) Iodic acid solution, containing 200 grammes per litre, prepared beforehand in order to allow the barium iodate, almost always present in the commercial acid, to subside. (2) Decinormal sodium thiosulphate solution. (3) A 20 per cent. solution of potassium iodide. (4) Fresh starch paste.

Antimony oxide (0·50 to 0·60 gramme) is placed in the apparatus used by Mohr or Fresenius in estimating iodine, and 20 to 25 c.c. of iodic acid are added, 10 c.c. of potassium iodide being placed in the tube condenser. On heating gradually to boiling, the iodine is liberated, absorbed by the iodide solution, and the iodine determined by titration with sodium thiosulphate. C. S.

Estimation of Metallic Iron in Iron reduced by Hydrogen. Dr. Schmidt. (*Vers. deutsch. Naturf. u. Aertze;* through *Rev. Chim. Analyt. appl.*, vol. vi. [3], p. 32.)—0·4 gramme of reduced iron is attacked in a 100 c.c. flask by 2 to 2·5 grammes of iodine (accurately weighed) in presence of 5 to 10 c.c. of water; the iron alone is dissolved under these conditions, leaving the oxide. One gramme of potassium iodide is then added and the liquid made up to 100 c.c., the excess of iodine being titrated with sodium thiosulphate. C. S.

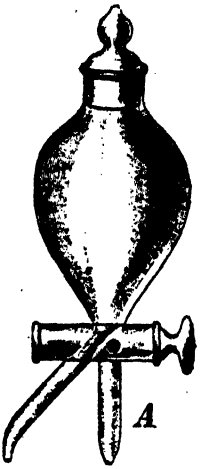
Contribution to our Knowledge of the Rare Earths. L. Haber. (*Monatsheft für Chemie*, xviii., 687.)—The author has studied the behaviour of the rare earths to chromic acid, potassium bichromate, sodium acetate and formate, and to tartaric, citric, and malic acids. The various reactions are summarized in the following table:

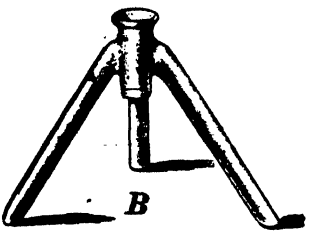
	Chromic Acid.	Potassium or Sodium Bichromate.	Sodium Acetate.	Sodium Formate.	Tartaric Acid.	Citric Acid.	Malic Acid.
Thorium -	Precipitates on boiling with difficulty and incompletely. Precipitate is orange-yellow and distinctly crystalline.	Precipitates easily from sufficiently diluted solutions on boiling. Precipitation is incomplete, and precipitate insoluble in water.	Precipitates from not too dilute solutions on boiling. Precipitate is white and crystalline.	Precipitates with difficulty on boiling. Precipitate is white and crystalline.	Precipitates easily, but incompletely, on boiling. Precipitate is white and flocky, and soluble in sodium acetate and formate.	Precipitates easily, like the preceding. Soluble in sodium acetate and formate.	Precipitates on boiling as a white, crystalline precipitate; soluble in sodium acetate and formate.
Zirconium	Precipitates moderately, easily, and completely on boiling. Precipitate orange-yellow and flocky.	Precipitates in a flocky condition, very easily on boiling.	Precipitates very easily and completely on boiling. Precipitate is white and flocky.	Precipitates very easily on boiling. Precipitate is white and flocky.	Precipitates incompletely in a fine flocky condition; soluble in sodium acetate and in excess of precipitant.	Precipitates easily in a flocky condition on boiling. Soluble in sodium acetate and in excess of precipitant.	Precipitates easily on boiling. Flocky; soluble in sodium acetate and in excess of precipitant.
Cerium -	Not precipitated.	Not precipitated.	Not precipitated.	Precipitates incompletely from concentrated solutions. Precipitate is beautifully crystalline.	Not precipitated.	Not precipitated.	Not precipitated.
Lanthanum	Not precipitated.	Not precipitated.	Not precipitated.	Precipitates incompletely from concentrated solutions. Beautifully crystalline.	Not precipitated.	Not precipitated.	Not precipitated.
Didymium	Not precipitated.	Not precipitated.	Not precipitated.	Precipitates incompletely from concentrated solutions. Beautifully crystalline.	Not precipitated.	Not precipitated.	Not precipitated.
Yttria - -	Not precipitated.	Not precipitated.	Precipitates with difficulty from concentrated solutions.	(?)	(?)	(?)	(?)

Thorium and zirconium are both characterized by the formation of basic salts with the organic acids; but whereas the thorium salts appear to be of constant composition, the zirconium compounds by continued treatment with water undergo a gradual change towards the condition of hydrate. H. H. B. S.

APPARATUS.

A New Separating Funnel. E. Spaeth. (*Zeit. für Untersuch. der Nahr. und Genussmittel*, 1896, 96.)—The funnel is provided with a two-way stopcock, the channels of which are separate and distinct from each other. One channel is straight and the other oblique, and each connects with a separate efflux tubulure, as shown in Fig. A. It is also provided with a stand, shown in Fig. B. In using the funnel it is obvious that, after the lower layer of liquid has been drawn off through the vertical channel, the upper layer can be run out through the other, which will be quite clean and uncontaminated. H. H. B. S.





MISCELLANEOUS.

The following article appeared in the *Times* of April 18, 1898:

"POLLUTED WATER AND SOMERSET HOUSE.

"(FROM A CORRESPONDENT.)

"After the experiences of Maidstone last winter, it might be expected that at least no obstacles would be placed in the way of the sanitary authority in dealing with all possible sources of danger within their jurisdiction. The public will, therefore, learn with astonishment that their efforts to do their duty by the community and to prevent the continued use of water from a dangerous source have been frustrated by the action of the local justices, with the assistance of two Government officials attached to the Inland Revenue Department. The whole case is so irregular, and so important to all sanitary authorities, that it deserves the widest publicity. The facts are briefly these:

"In Maidstone, as in most other towns, there are, in addition to the public water supply, a certain number of wells still in use. One of these, situated in Orchard Street, supplies four houses in that street which have not had the public water laid on. In the immediate neighbourhood fourteen cases of typhoid fever occurred in ten houses, distributed on all sides of the spot where the well is, and in dangerous proximity to it. The water, on being analysed by the

medical officer of health—who is also analyst to the county of Kent, and an exceptionally competent authority—showed signs of organic impurity, not to an alarming extent when taken by themselves, but more than sufficient to prove danger when taken in conjunction with the situation of the well and the known defective condition of the house-drains near it. The medical officer, guided by all the circumstances, condemned the water for drinking purposes, as every competent officer must have done in his position; but he did not recommend compulsory closure of the well, hoping to attain the same end more easily by persuasion. The wording of the Act makes it very difficult to obtain an order for compulsory closing except on chemical grounds, and in this case the chemical evidence was of less weight than the other conditions, and, apart from them, susceptible of a more favourable interpretation. However, the sanitary authority, actuated by a laudable anxiety to protect the town, applied for a closing order. It should be observed here that there is no reason why the well should not be closed, as the public water-supply is laid on in the street. The case came on for the first time on January 25, when it was adjourned for the defendant's chemist to attend. At the second hearing, on February 15, two chemists appeared for the defendant, and pronounced the water wholesome on chemical grounds, as they were quite entitled to do. The results of their analysis agreed with those of the medical officer; the difference lay in his interpretation, which was influenced by knowledge not within the reach of the other chemists. The magistrates then took a most singular step. They adjourned a second time, and ordered a sample to be sent to the Inland Revenue Department at Somerset House. At the third hearing, on March 15, a certificate was produced, signed by two officials in the Government laboratory, but not by the principal. The certificate, after giving the results of the analysis, which agreed in the main with those previously made, went on to say: 'From a consideration of all the results of the analyses there are grounds for stating that the water is not liable to contamination from the immediate neighbourhood of the well, and, although the water on account of its hardness could not be recommended for general domestic purposes, we are of opinion that its use for potable purposes is not likely to prove injurious or dangerous to health.' The magistrates thereupon dismissed the application, with costs against the sanitary authority. The public importance of the case lies in the fact that wells of this kind are one of the most dangerous and troublesome sources of disease with which sanitary authorities have to deal. It is difficult enough to get them closed at any time under the law, but if the analysts of Somerset House are to be made the arbiters—which is not provided by the law—it will clearly become impossible. Though doubtless highly competent in their own line, they have nothing to do with water, and still less to do with sanitation. By the Food and Drugs Act of 1875 the Inland Revenue Laboratory was appointed referee for the analysis of food and drugs in disputed cases, but water is expressly exempted by section 2 of the Act. Somerset House therefore, has no official or legal *status* in the present case. The terms of the certificate quoted above are a sufficient proof that it ought not to be allowed to usurp any such *status*. An analyst has a perfect right to say that a sample of water submitted to him is wholesome and fit to drink, but no one possessing an elementary knowledge of sanitary science would venture to certify, on chemical grounds, that a water is 'not liable to contamination,' or 'not likely to prove dangerous to health.' Those are questions the answer to which depends on the conditions surrounding the source of the water. A perfectly pure water may obviously be liable to dangerous pollution if the circumstances favour it. The greatest authorities have long ago laid down the axiom, which is now universally accepted, that chemical analysis can only be accepted as positive evidence of danger, not as negative evidence as to its absence. To use the words of the late Sir George Buchanan: 'The chemist can tell us of impurity and hazard, but not of purity and safety. For information about these we must go, with the aid of what the chemist has been able to teach us, in search of the conditions surrounding the water sources.' The same principle has been further laid down by the Local Government Board in relation to wells precisely similar to the one in question—'We must go beyond the laboratory for evidence of any drinking water being free from dangerous organic pollution.' When a water actually shows traces of organic impurity, it is an astonishing subversion of established principles for any responsible person to give a certificate of safety, not only in the present but for the future, on the strength of analysis alone and without taking the surrounding conditions into account. Considering what they are in the case of the Orchard Street well, one can only describe such a certificate as reckless. The danger to be apprehended is that specific pollution, which there is reason to believe has soaked into the ground, will gradually find its way into the well. The results of the three analyses confirm that danger. There is no discrepancy between them, but the two later ones indicate somewhat more impurity than that originally made, and point to a process of progressive pollution. The medical officer has recommended his committee to apply for the intervention of the Local Government Board, which is the proper authority to decide

the case; and meanwhile the action of the Somerset House analysts is to be brought up in the House of Commons immediately. The interference with the sanitary administration of the country by a Government department which possesses neither authority nor knowledge of the subject is wholly mischievous. In Maidstone alone there are several other wells which ought to be closed, but in face of recent proceedings it is obviously useless for the sanitary authority to take action."

[The following table, extracted from a report by Mr. Matthew A. Adams, F.R.C.S., F.I.C., of the case referred to, shows the local standard for unpolluted ragstone water, the results of the analysis of the water from the well condemned by Mr. Adams, the results of the analysis made by Mr. Gregory on behalf of the owner of the well, and the results of the analysis made by the chemists of the Inland Revenue Department. A full copy of the report made by the Government chemists is also appended.]

WATER ANALYSIS—REPORT.

	Local Standard for Ragstone Water Conduit Supply.	Adams. Sept. 17, 1897.	Gregory. Dec. 2, 1897.	Somerset House. Feb. 16, 1898.
Total solids	32·89	53·6	52·0	54·1
Loss on ignition	2·51	2·0	?	?
Chlorine	2·30	3·4	3·01	2·7
Nitrogen as nitrates ...	·466	1·14	1·840	1·81
Free ammonia	·005	·00	·02	·01
Albuminoid ammonia ...	·015	·05	·03	·064
Oxygen absorbed in ¼ hour	·008	·013	·0096	·0063
„ „ in 4 hours	·018	·018		
Hardness, total	17·4	23·1	33·6	34·0
„ permanent ...	6·5	15·6		20·0
Appearance in 2-foot tube ...	Clear p. blue	Greenish blue, rather dirty.		Clear, colourless
Smell	None	None		None
Phosphoric acid	Trace	Slight trace.		Trace
Nitrites				Trace

All results are given in grains per gallon, except free and albuminoid ammonia, which are in parts per million.

GOVERNMENT LABORATORY,
SOMERSET HOUSE, LONDON, W.C.

The sample of water referred to in your letter of the 16th ultimo, and stated on label to have been obtained from "Orchard Street," on "16th of February, 1898," was received here on the following day, securely sealed. The water was found to be colourless, clear, and odourless.

We hereby certify that we have analysed the water, and declare the results of our analysis to be as follows:

Albuminoid ammonia ... ... =	·0064	Parts per 100,000.
Free ammonia ... ... =	·001	
Oxygen consumed ... ... =	·009	
Total solids (dried at 212° F.) ... ... =	54·1	Grains per gallon.
Chlorides (stated as sodium chloride) ... ... =	4·45	
Nitrates (stated as nitrogen) ... ... =	1·81	
Nitrites ... ... =	trace.	
Total hardness ... ... =	34·0	Degrees per Gallon.
Permanent hardness ... ... =	20·0	

Judging from the figures shown under the terms "albuminoid ammonia" and "oxygen consumed," the water is regarded as of fair quality, and the results in these respects compare favourably with those obtained from samples of water drawn from the mains of the London Water Companies during the month of December. The proportion of nitrates is rather high, but having regard to the character of the mineral constituents of the sample, it does not indicate that the water is exposed to organic contamination.

The amount of chlorides for so hard a water is not regarded as excessive.

From a consideration of all the results of the analysis, there are grounds for stating that the water is not liable to contamination from the immediate neighbourhood of the well; and although the water, on account of its hardness, could not be recommended for general domestic purposes, we are of opinion that its use for potable purposes is not likely to prove injurious or dangerous to health.

As witness our hands this Eleventh day of March, 1898.

The Clerk to the Magistrates, Maidstone. (Signed) R. BANNISTER, G. LEWIN.

[On Thursday, April 21, a question was put in the House of Commons to the Secretary to the Treasury on the case referred to. The following is a report of the proceedings.]

HOUSE OF COMMONS,
Thursday, April 21, 1898.

Mr. HENRY J. WILSON asked the Secretary to the Treasury, in view of the fact that the chemical officers of the Inland Revenue are appointed referees in disputed cases of adulteration under the Sale of Food and Drugs Act, and that water is specially excluded from the operation of that Act under Clause 3, would he state whether it was in any way the duty of the chemical officers to undertake the analysis of water for sanitary purposes, and act as referees in disputed cases; and under what authority the chemical officers of the Inland Revenue recently analysed the water taken from a well at Maidstone, known as Prosser's Well, and in their report expressed the opinion that the water was not liable to contamination?

Whether, as a consequence of such report, the application to the Court to close the well was refused?

Was he aware that this well is in close proximity to houses in which no less than fourteen cases of enteric fever occurred during the recent epidemic?

And whether, seeing that the report was not signed by the chief of the laboratory, the action of these chemical officers was sanctioned by the responsible authority?

Mr. HANBURY: Under section 70 of the Public Health Act, 38 and 39 Vict., cap. 55, the magistrates are empowered, if they see fit, to cause water of which complaint is made to be analysed at the cost of the local authority. In cases of difficulty magistrates occasionally ask for the assistance of the Government laboratory, and it has been the practice for many years, when such assistance is asked for by the Court, to undertake an analysis of the water. In the present instance, the justices at Maidstone requested the assistance of the Government laboratory, and it was given in accordance with the usual practice. With regard to the latter part of the question, I understand that evidence as to the possibility of danger arising from the local surroundings of the well was given before the Court by the Medical Officer of Health, and that the magistrates, on consideration of all the evidence before them, decided to refuse the application. The action of the laboratory officers was not specially sanctioned by the Board of Inland Revenue in the particular instance. But these officers undertook the analysis at the request of the magistrates, in accordance with a practice which has for years existed with the approval of the Board.

Mr. JOHN ELLIS: Is the right hon. gentleman aware that the report which the officers drew up went far in excess of anything with which they were justified in dealing?

Mr. HANBURY: I think their report would go perhaps beyond their powers (hear, hear).

[The *Times* of April 19 draws attention to a somewhat similar case, reported in the ANALYST for October, 1893.]

THE ANALYST.

JUNE, 1898.

THE LATE LORD PLAYFAIR, G.C.B., LL.D., F.R.S.

We regret to announce the death, in his seventy-ninth year, of the Right Hon. Lord Playfair. Before he abandoned science for politics, Lord Playfair had a close connection with chemistry, which in his early youth he studied at the University of St. Andrews and at the Andersonian Institution, Glasgow, as well as under Graham at University College. He subsequently went to Giessen, where he became a pupil of Liebig, whom personally, as well as through his translations of the works of the great German chemist, he was instrumental in introducing to the British public. For some years he was engaged in industrial chemistry, and in 1843 was appointed Professor of Chemistry at the Royal Institution at Manchester, a post which he retained until, in 1846, in recognition of his services on a Royal Sanitary Commission, he obtained a Government appointment, as chemist to the Museum of Practical Geology. After a good deal of official work in connection with the 1851 exhibition, he was appointed Professor of Chemistry at the University of Edinburgh, a post which he held for thirteen years, during which time he took an official part in various public duties. Towards the close of this period he entered Parliament, and has ever since been chiefly known in connection with social, parliamentary, and political work, lying outside of the more strictly scientific pursuits of his earlier life. He was made K.C.B. in 1883, and created a peer in 1892. He was a founder of the Chemical Society, whose presidential chair he occupied many years ago.

Lord Playfair was elected an honorary member of the Society of Public Analysts in 1875, in recognition of the strenuous and successful exertions which he had made in the House of Commons for the improvement of the Sale of Food and Drugs Bill, which passed into law in that year, a similar compliment being at the same time paid to his then colleague in the House of Commons, Dr. (now Sir Charles) Cameron.

PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS.

The monthly meeting of the society was held on Wednesday evening, May 4, in the Chemical Society's Rooms, Burlington House, Piccadilly, the President (Dr. Bernard Dyer) being in the chair.

The minutes of the previous meeting were read and confirmed.

Mr. H. J. Lewin, analyst at the Royal Victoria Yard, Deptford, was proposed for

election as a member of the society; and Mr. F. M. Wharton, A.I.C., assistant to Mr. Chattaway, was proposed for election as an associate.

A paper was read by Dr. Matthew A. Adams, F.R.C.S., F.I.C., on "Water Supply in Relation to the Maidstone Epidemic."

WATER-SUPPLY IN RELATION TO THE MAIDSTONE EPIDEMIC.

By M. A. Adams, F.R.C.S., F.I.C.

(*Read at the Meeting, May* 4, 1898.)

The observations and experiments I have the pleasure of bringing under your notice to-night, however commonplace in themselves, cannot fail to have a special interest because of their association with, and bearing upon, an epidemic of typhoid unique for its magnitude in the annals of British medicine. As it will be assumed in that which is to follow that this epidemic was the result of the drinking of water poisoned by typhoid material, it is necessary in the first place that I should give at least a sketch of the facts that this assumption is based upon. For five-and-thirty years, during which I have resided in the town, Maidstone has enjoyed a reputation favourable for exemption from typhoid, for the death-rate from this disease during 1879-96 (the eighteen years of my occupying the position of Medical Officer of Health) has been only 121 per million, against the following rates for England and Wales and Kent respectively:

England and Wales { 1871-75 = 374; 1876-80 = 277; 1881-85 = 211; 1886-90 = 179 } *Kent* { 1871-80 = 330; 1881-90 = 180 }

Moreover, during the first seven and a half months of 1897 only five cases were reported, the last of these being an importation from Gibraltar, so that the news of a serious outbreak, which reached me whilst I was away on the Continent for my summer holiday, came as a complete surprise. The first cases to be notified were 2 on Saturday, September 11, next day 4 more were notified, and on Monday another, making a total of 7 in three days. This was the first warning of the coming storm. Inquiries were immediately set on foot to trace any possible connection with milk or other sorts of food. At this time no suspicion attached to the water-supply, but as on the 16th 27 fresh notifications were received, then the water began to be suspected. On the 17th 26 more cases occurred, making altogether 64 notified in six days. These having been plotted out upon a spot map, it soon became apparent that the incidence of the disease was confined to certain parts of the town, practically to the entire exclusion of all districts excepting those that were supplied by a particular branch of the public water service.

Maidstone derives its water-supply from three sources, as follows:

1. From chalk springs at Cossington.
2. From chalk springs at Boarley.
3. From greensand springs at Farleigh.

The affected districts coincided absolutely with the Farleigh area of supply, and as time went on this feature in the distribution of the disease became more and more noticeable, as it were dissecting the town street by street and house by house, in accordance with the distribution of the Farleigh water—so accurately, indeed, that as a fact one small district, to which I shall presently refer as the "Special Area," comprising 338 houses, which in the first instance we had been led to believe was supplied with Cossington water, by the incidence of this disease was discovered in reality to have been supplied for the time being, through an oversight, with Farleigh water. Naturally, as time went on, a certain amount of spreading took place into other parts of the town by the casual drinking of the Farleigh water by the inhabitants of the other two water areas and by personal contagion, but the main features of its territorial distribution were maintained to the end, as is demonstrated by the following completed table of incidence:

Cossington Supply.

District.	Houses.			Persons.		
	Number.	Invaded.	Per cent.	Population.	Attacked.	Per cent.
To West Maidstone	585	12	2·05	2,925	19	0·64
To East Maidstone	86	7	8·14	430	10	2·32
Total	671	19	2·8	3,355	29	0·86
Boarley Supply.						
To West Maidstone	—	—	—	—	—	—
To East Maidstone	1,410	63	4·46	7,050	69	0·97
Total	1,410	63	4·46	7,050	69	0·97
Farleigh Supply.						
To West Maidstone	1,618	435	26·8	8,090	673	8·32
To East Maidstone	1,960	547	27·9	9,800	776	7·92
Special Area	338	95	28·1	1,690	134	7·92
Total	3,916	1,077	27·5	19,580	1,583	8·08
Grand Total	5,997	1,159	19·32	29,985	1,681	5·60

From which we learn that there are 5,997 houses in Maidstone supplied by the Water Company—671 of these with the Cossington water, 1,410 with the Boarley water, and 3,916 with the Farleigh water. Of the 671 in the Cossington area, 19 (= 2·8 per cent.) were invaded; of the 1,410 houses in the Boarley area, 63 (= 4·46 per cent.); and of the 3,916 houses in the Farleigh area, 1,077 (= 27·5 per cent.) were invaded. Then as regards persons: out of a population of 3,355 in the Cossington area, 29 (= 0·86 per cent.) were attacked; of the 7,050 in the Boarley area, 69 (= 0·97 per cent.); whilst of the 19,580 in Farleigh, 1,583 (= 8·08 per cent.) were attacked.

Furthermore, when we break up the districts of supply into sub-districts, we notice the rates of invasion of houses and attacks upon the respective populations are

as irregular as can be, so far as they relate to the Cossington and Boarley areas, but just the reverse in their relation to the Farleigh area. For instance, the rates of invasion of the houses in the Cossington sub-districts and the Boarley district were 2·05, 8·14, and 4·46, whereas in the Farleigh district they were 26·8, 27·9, 28·1. Mark you, the *special area* which (as already said) was originally supposed to have received its supply from the Cossington springs by the rate of the invasion of the houses led to a special investigation, whereupon it was discovered that, owing to a temporary exigency of the service, this special area for the time being was supplied from the Farleigh springs, with which service, as you see by the rates, it exactly corresponds. In the same way the same contrasts are manifest in regard to the persons attacked; whilst in the Cossington and Boarley districts the rates of attack vary tremendously (0·64 per cent., 2·32 per cent., and 0·97 per cent.), in the Farleigh they are practically identical (8·32 per cent., 7·92 per cent., and 7·92 per cent.), clearly showing that whilst the agent of mischief in the Farleigh district had been equal and uniform in its operation, such had not been the case in the Cossington and Boarley districts. In other words, the cause of the mischief had been the *habitual* drinking of the Farleigh water by the inhabitants of the Farleigh district, and the *casual* drinking of the Farleigh water by the inhabitants of the Cossington and Boarley districts.

I think I have now said enough to justify the suspicion which fell upon the Farleigh water, and, the suspicion having been aroused, samples were collected for analysis on September 19, 20, and 25. I may here remark that this Farleigh water is itself a compound water, the joint produce of a number of springs derived from the Hythe beds of the lower greensand situated right and left of the river Medway, which is their natural drainage outlet. Composed simply of subsoil water, these springs emerge at the junction of the greensand with the underlying Atherfield clay, and have been in use for the public water-supply for very many years. In the pursuit of my official duties I have made periodical analyses of the joint product of these springs for a space of twenty-five years, but until the time of the epidemic never of the individual springs separately. These periodical analyses have shown, in later years at all events, very great constancy of composition, so that we had come to regard the Farleigh source as thoroughly trustworthy. We have still another public water-supply in Maidstone, the property of the town, as distinct from the property of the Water Company, called the Conduit Supply. This comes from a source exactly similar to that of the Farleigh springs. A long series of periodical analyses of this water shows its composition to be practically invariable. For this reason I have made the Conduit water serve as my "*local standard*," for purity of greensand, or, as we say locally, Ragstone water, to which all waters in our neighbourhood derived from that formation, if pure, should strictly conform. As all the conclusions relating to the analyses which I am now about to submit to you are based upon a comparison with this local standard, I have inserted the figures relating thereto in the first column of the table of results of the analyses. Before entering upon a consideration of the figures of the analysis, it is proper I should state that, to the best of my judgment, the poisoning of the water commenced about August 17, and terminated about September 25; that it was at its worst during the last four

days of August and the first four days of September; and, further, that, judging by the dates of attack, and making allowance for the period of incubation, the violence of the epidemic had long passed when the first sample of water was collected.

Analyses of the Farleigh Waters.—The periodical analyses, which had formerly been made once a fortnight, for reasons of economy were cut down by the borough authorities to once a quarter, and the last occasion on which the Farleigh water was analysed previously to the outbreak was on June 25, with the following results:

	Standard.	June 25.	
Total solids	32·890	33·900	grains per gal.
Chlorine	2·300	2·500	,, ,,
Nitrogen as nitrates...	0·466	0·570	,, ,,
Free ammonia	0·005	0·010	pts. per mill.
Albuminoid ammonia	0·015	0·010	,, ,,
Oxygen absorbed in a quarter of an hour	0·008	0·009	grains per gal.
Oxygen absorbed in four hours ...	0·018	0·012	,, ,,
Total hardness	17·400	16·800	,, ,,
Permanent hardness...	6·500	8·000	,, ,,

The main difference from the standard was 1 grain per gallon in excess of total solids, 0·2 of a grain chlorine, and 0·1 nitric nitrogen. Though the total hardness was a shade less, the permanent hardness was 1½ degrees more, in consequence of the increased nitrates. From a consideration of these figures, I think we shall agree that the variations from the standard were but trifling, and in no way calculated to create alarm. Unfortunately, the quarterly arrangement was the reason of no further analysis being undertaken until after the outbreak—in fact, not until September 19, when suspicion had fallen upon the water. Then, of course, careful inspection of all the springs that contributed to the Farleigh supply was immediately set on foot, and samples collected for analysis from each of them. Meanwhile, the most suspicious of the springs, so far as local appearances went, were cut off. The results of the analyses of these various samples, together with the corresponding figures of the local standard, are set forth in the table on pages 154, 155, a comparison with which shows the *total solids* to be in excess in almost every case, sometimes to a considerable amount. The *chlorine*, though never a large item, shows excess in a few instances, but it is as often low as it is above the standard. *Nitrogen, as nitrates* and nitrites, in every case but one shows excess, often very great excess. *Free ammonia* was either absent or exhibited a mere trace. *Albuminoid ammonia*, in all but seven instances, was an inconspicuous figure; in five cases it showed in considerable, but never in remarkable, excess. *Oxygen consumed* was never in substantial excess. The *hardness*, especially the *permanent hardness*, was practically always in excess, except in the case of the Ewell springs. *Phosphoric acid* showed considerable or large excess in only about four instances.

Physical Appearance.—The Tutsham-in-Fields water alone was conspicuously at fault. In about three other instances there was noticeable greenish opacity, but nothing to attract particular attention. The appearance of the *residue* both before and after ignition in many instances gave occasion for remark. By far the most

important and most frequent variation from the standard was the excess of nitrogen as nitrates, and I invite your special attention to this feature of the analyses for reasons which will be more apparent by-and-by.

For the present I must leave the chemical aspect of the question, and ask you to follow me in the consideration of certain meteorological events that appear to have had a most important bearing upon the cause and method of the pollution of the water. For many years past I have recorded the daily fluctuations in the level of the subsoil-water; these observations led me to discover the occurrence of certain unusual features concerning the fluctuations that occurred during last August, exactly corresponding with the occurrence of the typhoid outbreak. These I will endeavour to make clear to you. In diagram No. 1 three sets of facts are exhibited by a graphic method drawn to scale. In the lower part we have the daily record of the subsoil-water in feet; upon this, in black, is seen the rainfall in inches. At the upper part of the diagram are recorded the cases of typhoid referred to the dates of attack.

For the present it is my desire that your attention be confined to the lower part of the diagram, to the records of the subsoil-water levels and the rainfall, and the points I more particularly wish to discuss with you are: the general contour of the ground-water level, the factors that are instrumental in raising or depressing the level, and the interpretation of certain abnormalities which, as we shall see, exactly coincide with, and, as I shall endeavour to show, were instrumental in causing the epidemic. In the first place you will observe that the ground-water level was at its highest in February and at its lowest in November, that it exhibits, speaking generally, a more or less gentle and uniform fall during the intervening eight months, with certain interruptions about which I shall have more to say by-and-by.

The rainfall, which, by the way, was remarkably scanty—in fact, the lowest recorded since 1870, being only 19·73 inches, against a twenty years' average of 25·9—was distributed in such a manner that a great part of June, the whole of July, and the first six days of August—the hottest part of the year—were almost rainless. Of course, the level of the subsoil water is in great measure, but by no means wholly, dependent upon the rainfall. Two other factors have great influence upon it, viz., evaporation, caused by sunshine and wind, for one; and plant-life, causing absorption and dissipation, for another. A critical examination of this and similar diagrams relating to other years affords evidence that the water-level is usually at its maximum height during the early months of the year, and commences its descent in April, after the vernal equinox, when plant-life begins to be active; and the sinking continues through the summer solstice so long as light abounds and plant-life continues vigorous, even in spite of rainfall, until the autumnal equinox, when the earth enters the winter solstice at the end of September. Then, or at the beginning of October, a rise usually sets in, though such was not the case in 1897, for we find the descent continued till November 28—but last year was altogether a most exceptional year. Of course, this order of events is liable to a temporary interruption, and the curve is subject to more or less distinctly marked irregularities by the occurrence of rain, and it is to the minor details of this matter I would now ask your special attention. Let us begin with the year. Between January 4 and 10 there was a pretty consider-

able rainfall, and its effect upon the water-level can be traced in the rise commencing on the 8th; during the last two days in January and the first five days of February a large mass of water fell, and the consequent effect is far more obvious; up went the subsoil-water till it reached its maximum on February 11; a small, sharp and temporary rise followed a sudden and copious rainfall during the first two or three days of March, but, in spite of the fact that March, with seventeen days of rain, proved a wet month, the sun having gained power, a check was set upon the further rise of the ground-water. During April, its fourteen days of showery weather notwithstanding, down went the subsoil-water, chiefly owing to the vigour of plant-life, so that by the end of May, though there had then occurred a whole week of heavy rain, the water-level continued to sink rapidly. On June 8 there came a very heavy fall (nine-tenths of an inch), all within a few hours, but, mark you, only a slight impression is produced. The rest of June, and practically the whole of July, and the first six days of August, were rainless; on August 7 and 8, two most momentous days for Maidstone, something less than half an inch of rain fell, but, as a consequence, behold the prodigious rise in the subsoil-water level. On the 15th and 17th there was more fall, and the level continued to rise. On the 18th still more rain, but mark that, instead of a further rise, the ground-water began to descend, and continued to do so in spite of more rain on the 20th, 24th and 26th, the descent being as rapid as the previous ascent had been, even though the rainfall on the 26th, on one day alone, was greater than that which fell on the 7th and 8th together, when such an amazing effect had been produced. During the last three days of August and the first day of September about three-quarters of an inch of rain produced another considerable, though less conspicuous, rising, which, like its precursor, soon subsided. After this a marked rise could not be maintained even by the considerable rains of September 6, 18 and 19, nor restored by one of the heaviest rains of the year, on the 29th.

Now let us look into the explanation of all this: Geologically, Maidstone is situated at the junction of the lower greensand with the Atherfield clay, where the constituents of the two rocks mingle, so that our soil and subsoil consist of sandy or clayey loam, according to the circumstance of the exact situation. At this point it is essential I should remind you of a remarkable property exhibited by clay rocks. I allude to the circumstance peculiar to clay—that it expands in the moist condition and largely contracts when dry; moreover, when greatly desiccated, it becomes hard as a clinker; as a consequence, in times of drought, under this latter condition clay soils are liable to become fissured; and as a fact, established by personal observation, during last summer's drought I found wide cracks into which a stick could be thrust 3 feet deep into the subsoil without reaching bottom; how deep the cracks went I have no idea. With this condition of things, it is obvious that the rains of August, instead of penetrating into and being absorbed by the soil and subsoil could, and would tend to find a direct road through the cracks straight away into the subsoil springs, accumulating in the first instance in the cracks between the desiccated fragments without being absorbed into the mass of the soil, thereby quickly, though temporarily, raising the level of the ground-water, that is to say, in the spaces between the fragments of the cracked soil; but so soon as absorption of the water into the

mass of the soil took place, the adventitious rise would give way to subsidence, and thereby the water level would sink as quickly as it had risen. About a fortnight later these phenomena were repeated, with this difference: we find that though the rainfall was much greater, the effect was much less, because the surface induration of the clay fragments having already been overcome by the earlier rains, capillary absorption had in a measure been restored to its normal condition. In connection with this branch of the subject there are many interesting points of detail that one would like to dwell upon, such as a comparison of the various effects of similar rainfalls in different conditions of the soil. For example, compare the rain of August 7 and 8 with the almost exactly similar fall on September 18 and 19, producing in the earlier case a tremendous rise, because of the dry, cracked state of the soil—in the later case, when the soil was moist and the cracks had closed up, producing no sensible effect at all. But time presses, and we must pass on to a consideration of the upper part of the diagram. There we have depicted the cases of typhoid according to their dates of attack. In order to make the occurrence of the two sets of events—that is to say, the pathological and meteorological; the typhoid and the movements of the ground-water—comparable, a proper allowance must be made for the period of incubation of typhoid: fourteen days is reckoned an average period, but it varies considerably. In addition to the fourteen days for incubation, a day or two must be allowed for the water to get to the consumer, so that in the diagram I have set back the typhoid manifestations sixteen days. This should bring the two sets of events approximately into relation as regards time. It is practically certain that in the mass of cases recorded there must be some with short incubation; of course, it is impossible to distinguish between these and those of average incubation, so that the few scattered earliest cases as they appear in the diagram are those having short incubation, and consequently set too far back. Be that as it may, except for the two or three earliest cases, the commencement of the epidemic exactly coincides with the summit of the first rise, and the acme of the great mass of the disease coincided with the summit of the second rise. These coincidences are more than curious; surely they stand related to one another as cause and effect.

Allow me to recapitulate the facts: We have a subsoil-water stored beneath a loamy clay, situated in a highly-cultivated district, where heavily-manured crops are grown, and where many people are employed in the cultivation and gathering of fruit and hops. The surface is necessarily bestrewn with effete organic matter, and exposed to casual pollution by the workpeople employed. An unusual drought occurs, giving rise to cracks in the soil and subsoil. We have physical evidence of the rapid passage through to the subsoil water-level of the surface-water derived from the rains of August 7 and 8. Chemical analyses bear evidence that the ordinary natural processes of purification are in abeyance. A six days' pause from rain follows; polluting specific matter is retained in the cracks of the soil and subsoil hot from the summer's sun; incubation of the putrefying and specific organisms takes place in the dark, moist subterranean recesses, the cultivation process being promoted by the showers that fell on the 16th, 17th, 18th, 20th. Then comes another pause, during which an abundant crop of organisms is produced, followed in six and more days later by heavier rains, which carry the pathogenic products into the ground-water, out by the springs,

straight away into the public mains, and, as a consequence, the epidemic immediately follows.

It appears to me the chain of causation and effect is complete. I should like to make a further use of these reflections. We all know what a difficult and responsible problem the interpretation of a water analysis for sanitary purposes often is, and anything that will help to a rational understanding of the natural causes of dangerous pollution is sure to be welcome. Moreover, if by an application of the rationale we can lead up to a natural generalization, it cannot fail to be of some use. Now, the diagram before you exhibits an ideal section of soil and subsoil, such as we have been contemplating. The surface is supposed to have been polluted by the deposit of effete animal matter, and the idea is to represent in outline the natural processes concerned that tend for and against the purity of the water that accumulates and flows beneath. At the outset, I would remind you that three distinct sets of operations are engaged in the protection of ground-water against organic pollution. First, there is the action of putrefying organisms; second, the action of nitrifying organisms; and third, the action of plants. Crude complex organic matter is first broken up into simpler organic matter; this is then mineralized into nitric, nitrous, and carbonic acids, and finally the nitrates are removed by plants. When these three operations are fully accomplished, water, however much polluted in the first instance, in the end is rendered pure and wholesome. In the main, the several operations are executed at successively deeper situations in the soil, and for convenience we may contemplate three distinct zones, each the seat of its appropriate function. The uppermost we may designate the "danger zone"—where the putrefying organisms are at work, converting the crude animal matter into leucine, tyrosine, toxalbumin, etc. The next we may style the "risky zone," where mineralization goes on, but where conversion is not complete, where the roots of plants take up the mineralized products, but also where the harmless and the poisonous products mingle. Beneath this we come to the "safe zone," where the water it contains is found to have been purified not only from all organic matter, but also more or less completely of all of its nitrogenous products. In Nature, of course, the situation of these zones is not fixed, but varies according to the exigencies of the climate, as I have attempted to portray in the diagram. It follows, therefore, if the foregoing propositions are correct, that the presence of an abundance of nitrates in subsoil-water in most cases is significant of risk, because it shows the water to be derived from the "risky zone," and implies the possibility of its coming from the "danger zone," and the certainty that it is, at least in an unrenewed condition, charged with the products of oxidized organic pollution. Remember we are contemplating subsoil-water; and I would here remind you that there is nothing to show that pathogenic live organisms are acted upon by the nitrifying organisms. For aught we know to the contrary, these latter only act upon dead organic matter; and so far as cholera and typhoid organisms are concerned, such protection as the soil can afford is rather of the nature of filtration than destruction. I now come to the last point I wish to discuss with you; it is one that has arisen out of the ground-water observations. We are all familiar with the circumstance that disorders pro-

pagated by the fæcal pollution of water, such as cholera and typhoid, are very commonly, though not always, liable to be preceded by what is called "*premonitory diarrhœa.*" This association, because of its uncertain occurrence, seems to be due to some, as yet undiscovered, extrinsic cause. In our case the epidemic *was* preceded by premonitory diarrhœa. Suddenly, before the earliest typhoid attacks occurred, the town was flooded by complaints of violent diarrhœa (see diagram No. 2) of quite a different sort from ordinary summer diarrhœa—in fact, more like that produced by poisoning. I have endeavoured to obtain accurate information concerning these occurrences, and certain of my medical friends have furnished me with particulars which show a correspondence as respects time between the symptoms of this illness and the first rising of the subsoil-water; but they also show that as the typhoid epidemic advanced the diarrhœal attacks subsided, from which it is but natural to infer that the water drunk by the people of Maidstone contained, temporarily only, a poisonous material capable of producing diarrhœa. Now, I suggest that toxalbumin was the cause, and that this material, having accumulated on the surface of the soil and within the danger zone, was washed down by the early rains of August in an unaltered condition through the risk zone without being nitrified, and so into the water that was being drunk during the early and middle part of August. Having regard to the physical condition of the soil and subsoil at this time, it seems quite possible; this material being soluble, we can readily understand that it would be easily and quickly washed through the subsoil, and that its effects would be spent in the early stages of the epidemic. On the other hand, the typhoid bacteria, being particulate and probably few at the start, might lodge in the dark recesses of the warm subsoil, there multiply by incubation during the interval between the two uprisings of the ground-water, and eventually be flushed forward by the rains that produced the second rise. This, it appears to me, is a reasonable explanation of the order and character of the events, of the premonitory diarrhœa, and the subsequent explosive outburst of typhoid. Though I have no physical evidence to substantiate it, this theory accurately fits in with the observed facts. The difficulty of obtaining experimental evidence, I imagine, would be very great, nevertheless; and I think you will agree with me the problem is of sufficient interest to deserve investigation from a chemical as well as an etiological point of view.

In this record of my experiences you will readily find subjects for discussion; in order, however, to give a practical turn to the matter, allow me to gather together in the form of propositions some of the principal points:

(1) That the recorded facts unmistakably inculpate the Farleigh water with the cause of the epidemic, and that the result was brought about by the unusual meteorological circumstances in the manner suggested.

(2) That the medical history of the epidemic indicates the presence of two sorts of poison in the Farleigh water, the one of a chemical toxic nature causing the "premonitory diarrhœa," another of a biologic nature, causing typhoid.

(3) That the most conspicuous features of the chemical analyses are (*a*) a large excess of nitrogen as nitrates, (*b*) a comparatively small excess of albuminoid ammonia, (*c*) a remarkably small amount of oxidizable matters.

(4) That this occasion affords another illustration of the impossibility of formu-

lating an artificial standard for the chemical constitution of potable water that can serve as a basis upon which to found a correct judgment as to its sanitary wholesomeness without other considerations being taken into account.

(5) That a better, simpler, and more natural basis is attained by the establishment of local standards.

(6) For this purpose, especially in the case of public water-supplies, it is essential that frequent analyses be made at short intervals, at all seasons, with or without bacteriological observations.

(7) That continuous observations should be made and records kept respecting the meteorological phenomena, especially the movements of the subsoil-water and the rainfall, these being desirable in every case, but absolutely necessary where the supply is derived from the subsoil.

(8) That apart from ordinary considerations due to the natural mineral constituents, a water that is of stable composition is wholesome; on the other hand, instability of composition, whatever the fluctuations may be due to, denotes risk.

(9) For the reasons adduced, the presence of nitrates or nitrites among the mineral constituents, unless there be some local geological explanation thereof, is indicative of risk—past, present, or future.

(10) That it is desirable that research should be directed to the chemical and physiological processes associated with filtration; among other things, to determine what happens to living pathogenic organisms. Are they attacked and destroyed under any, and, if so, what, circumstances?

In bringing this paper to a close, I must apologize for having spent so much time over the physical facts; but, if I am not mistaken, they lead to considerations that have not received the attention they require. According to my view, it has been too much the custom to look upon the analysis of water as having for its sole object the detection of pollution dangerous or injurious to health, whereas it ought to be the chief function of the sanitarian, whether he be chemist, medical officer, or water engineer, to prevent water from becoming dangerous or injurious, to anticipate pollution, and give warning of the approach of such. Surely the first step in the realization of this purpose is to ascertain the mechanism, so to speak, of its occurrence. This communication is intended as a contribution to that end, and, if in any sense successful, I hope it may stimulate to further observations, and perhaps assist in the interpretation of the chemical results of the analysis of water for sanitary purposes.

Discussion.

The President said that all of those present had probably to some extent familiarized themselves with the general subject of the Maidstone epidemic and its relation to water-supply through the abstracts of the reports of Dr. Adams and Dr. Washbourn which had appeared in the press. He was sure, however, that this paper would give them a much clearer and more instructive idea of the subject. The paper, no doubt, raised some debateable points; but however different individual opinions on some of the points might be, it would be generally agreed that Dr. Adams deserved hearty thanks for the very lucid way in which he had presented the facts

and views set forth in the paper, and that he had rendered very great service to the town of Maidstone by his work in the matter. The connection between the rainfall, the subsoil water-level, and the outbreak of the epidemic had been very lucidly demonstrated, and the chain of evidence all through seemed to be complete. The latter part of the paper, however, contained matter which at the present time might be regarded as almost new. Formerly the general opinion was that the danger from drinking polluted water was due actually to poisonous organic matter. Gradually that was given up, and the dangers attributable to drinking polluted water had been generally recognised to be due, not to dead organic matter, but to living organisms, which exercised pathogenic functions within the system. According to the view which Dr. Adams put forward, it was suggested that there was something in the older theory, after all, and that actually poisonous substances (presumably produced originally by micro-organisms, but already existing in the water) might be introduced —"in bulk," as it were—into the human system, instead of being developed by the bacteria after the consumption of the water. If this were really the case, one would expect to get some evidence of it in the shape of a rise in organic nitrogen; and for the examination of the view held by Dr. Adams, it was certainly unfortunate that there was not an analysis of the water at the earlier period, during which these substances might have been present, and when it could have been seen if there was an excessive quantity of albuminoid ammonia. But thanks to the zealous desire of the Maidstone Corporation to exercise small economies in order to keep down the rates, they were deprived of what might have been valuable information in the examination of this particular theory. What Dr. Adams had said with regard to local standards was most important, and also what he had said as to the very great value of nitrates as an indication of pollution, the latter point being noteworthy in view of a recent matter in which Dr. Adams had been concerned. Dr. Adams had lately condemned a well at Maidstone which was shown by chemical analysis to be grossly polluted, and which was close to drains in the neighbourhood of several typhoid-infected houses. The magistrates sought confirmation of Dr. Adams's opinion, in view of its being challenged, by an appeal to the Government chemists, who in their report pointed out that the water as to albuminoid ammonia and oxygen absorbed compared favourably with water drawn from the London water companies, but also that it contained 1·8 grains of nitric *nitrogen* per gallon (equivalent to nearly 7 grains of N_2O_5). They stated, certainly, that this proportion of nitrates was "rather high," but that, having regard to the character of the mineral constituents of the sample, this did not indicate that the water was exposed to organic contamination; and they expressed their opinion that the use of the water for potable purposes was not likely to prove injurious or dangerous to health. A comparison of the water with the "local standard" for unpolluted water in the neighbourhood would surely have rendered such a report impossible. Dr. Adams had said nothing in the paper about the bacteriological side of the investigation of the Maidstone epidemic; but Dr. Washbourn was present, who had worked side by side in the matter with Dr. Adams, and any remarks which he might make from this point of view would be listened to with interest.

Dr. J. W. Washbourn said that he thought it would be generally accepted by

the Society that this epidemic was produced by the Farleigh portion of the Maidstone water-supply. The evidence which Dr. Adams had brought forward in favour of that view was most conclusive. Chemical analysis had shown beyond question that the water had become polluted in some way or other with organic material, and the only question likely to be discussed in this connection was as to whether the organic material was of a vegetable or animal nature. With regard to the bacteriological examination, he might say that an examination was made of a large number of the springs making up the Farleigh supply. The first question to decide was whether the water contained any typhoid bacilli. The result of the examination in this direction was entirely negative, and this he attributed to two facts. In the first place the examination was, as had been pointed out by Dr. Adams, undertaken at a time when it was very likely that the typhoid bacilli had disappeared: and in the second place, the examination was not commenced until the epidemic was at its height, by which time it was very likely that the pollution had passed off. As the incubation period of the disease was about fourteen days, the examination ought to have been made at least a fortnight, if not three weeks, earlier. That was probably one reason for the negative nature of the results obtained. The other reason was no doubt the fact that the methods at present available for detecting the typhoid bacillus in contaminated water are very imperfect. He did not know of a single instance in which water suspected of conveying typhoid fever had been definitely proved to contain the typhoid bacillus. On several occasions it had been stated that typhoid bacilli had been found in drinking-water, but most of these examinations were made at a time when the methods adopted for distinguishing the typhoid bacillus were not sufficient to distinguish it from other allied bacilli. The best instance in which an examination with the object of detecting the typhoid bacillus seemed to have been carried out according to modern methods was that of experiments made by Remlinger and Schneider, who found typhoid bacilli, or at any rate bacilli having the characters of the typhoid bacillus, in a large number of samples of water and soil. In fact, the very large number of instances in which they found the typhoid bacillus looked suspicious, and the account given in the journal of the Pasteur Institute of the character of these bacilli was not sufficiently detailed to enable definite conclusions to be drawn. The next step in connection with the Maidstone water was to examine and enumerate the bacteria present. The number of bacteria found in water was of itself not of very great importance, though he thought that if a water was constantly found to contain a large number of bacteria, it might be taken as certain that it was polluted, not with animal matter necessarily, but with material of some kind from the surface. The surface of the soil contains a large number of bacteria, but the further down one goes, the fewer one finds, until the deepest layers are found to contain no bacteria at all. If a water was pure, it would contain very few bacteria, but a water might contain only a few bacteria and still be liable to contamination. On the other hand, a fairly large number of bacteria might be found in a water which was not polluted. Water that had been stagnant for some time in a well would, when it was first pumped up, contain a large number of bacteria, the fact of the matter being that the bacteria had sunk to the bottom, forming a sediment which would be drawn up by the first pumping. In judging the purity of a water from the

MAIDSTONE WATERWORKS COMPANY'S EAST FARLEIGH SUPPLY.

Typhoid Epidemic, 1897.

RESULTS OF CHEMICAL ANALYSES.

Source.	Local standard.	Collecting-Tanks, all sources, Sept. 19.	Tutsham-in-Field, Sept. 19.	Ewell Injection Tank, Sept. 19.	All sources, Public Hospital, Sept. 20.	Catch-pit No. 1, S.E.R., Sept. 20.	Catch-pit No. 2, S.E.R., Sept. 20.	Catch-pit No. 3, S.E.R., Sept. 20.	Catch-pit No. 4, S.E.R., Sept. 20.	Catch-pit No. 5, S.E.R., Sept. 20.
	0.	1.	2.	3.	4.	5.	6.	7.	8.	9.
Total solids	32·89	34·8	39·5	23·2	34·3	40·7	40·4	45·1	45·9	42·7
Loss on ignition ...	2·51	4·4	3·8	1·5	4·0	4·6	5·2	3·5	2·7	0·8
Chlorine ...	2·39	2·5	2·5	2·0	2·5	2·1	2·2	3·2	3·3	3·2
Nitrogen as nitrates and nitrites ...	·466	·98	·71	·70	·84	1·52	1·02	1·14	1·14	1·14
Excess of ditto, ditto. in per cent. upon the local standard ...		+110%	+52%	+50%	+80%	+226%	+119%	+144%	+144%	+144%
Free ammonia ...	·005	·01	Nil.	Nil.	Nil.	Nil.	Nil.	Nil.	Nil.	Nil.
Albuminoid ammonia	·015	·05	·13	·07	·01	·02	·01	·10	Nil.	·03
Oxygen ¼ hour ...	·008	·004	·012	·005	·005	·003	·005	·005	·008	·010
Oxygen 4 hours ...	·018	·022	·023	·017	·018	·021	·017	·020	·027	·032
Total hardness ...	17·4	15·8	18·7	12·7	15·6	19·2	20·0	21·3	20·4	21·2
Permanent hardness	6·5	·75	11·3	6·3	7·8	10·1	8·2	10·5	9·6	8·2
Two-foot tube ...	Clear pale-blue.	Clear greenish-blue.	Green, very turbid.	Clear green.	Clear bluish-green.	Clear bluish-green.	Clear bluish-green.	Clear greenish-blue.	Clear bluish-green.	Green, slightly turbid.
Phosphoric acid ...	Trace.	Slight trace.	Moderate trace.	Slight trace.	Moderate trace.	Slight trace.	Slight trace.	Moderate trace.	Trace.	Slight trace.
Smell ...	None.	None.	Slight.	None.	None.	None.	None.	None.	None.	None.
Appearance of residue on ignition	White.	Slightly blackens.	Blackens and fuses.	Blackens.	Blackens, and slight fusing.	Slightly blackens and fuses.	Slightly blackens and fuses.	Blackens and slight fusing.	Moderately blackens.	Moderate blackening and fusing.

Little Church. Catch-pit No. 6, S.E.R., Sept. 25.	Big Church. Catch-pit No. 7, S.E.R., Sept. 25.	Church End. Catch-pit No. 8, S.E.R., Sept. 25.	Hospital Wood. Catch-pit No. 9, S.E.R., Sept. 25.	Underbank. Catch-pit No. 10, S.E.R., Sept. 25.	End Spring. Catch-pit No. 11, S.E.R., Sept. 25.	Engine-room, all sources, less Tutsham, Sept. 22.	Tutsham-in-Field, Oct. 19.	Church End, No. 8 Catch-pit, Oct. 19.	Ewell Injection Tank, Oct. 19.	Culvert between Filter-Beds and Old Pump, Oct. 28.	Ewell supply, Air Pipe, Oct. 29.	Tutsham-in-Orchard, Nov. 16.
10.	11.	12.	13.	14.	15.	16.	17.	18.	19.	20.	21.	22.
41·1	39·6	41·5	36·9	45·3	49·7	32·8	38·5	38·6	22·9	27·3	23·1	34·8
2·9	2·7	4·9	2·3	3·5	3·5	2·1	4·9	3·0	1·9	2·7	3·2	2·6
3·0	3·2	2·2	2·4	2·9	2·5	2·7	2·1	2·2	1·8	1·9	1·8	1·9
·57	·64	·83	·64	·83	·81	·86	·81	·89	·62	·57	·38	·68
+22%	+37%	+78%	+37%	+78%	+73%	+84%	+73%	+90%	+33%	+22%	Nil.	+45%
Nil.	Nil.	Nil.	Nil.	Nil.	Nil.	Nil.	·01	·03	Nil.	·01	Nil.	Nil.
·01	Nil.	·01	Nil.	·02	02	·01	·17	·11	·02	·13	·02	·02
·006	·005	·009	·008	·013	·009	·005	·010	·007	·007	·011	·004	·003
·015	·015	·017	·015	·017	·019	·015	·020	·020	·010	·027	·008	·012
19·1	18·9	19·3	17·2	21·2	23·4	16·7	22·0	20·4	14·0	14·0	12·9	18·5
8·8	8·9	8·8	8·4	9·9	10·6	7·2	8·9	11·1	5·5	5·7	5·3	8·7
Clear bluish-green.	Clear bluish-green.	Clear green.	Clear green.	Clear green.	Clear green.	Gray or dirty green.	Opaque brown, very dirty and bad.	Blue-green, with suspended matter.	Pale clear blue.	Turbid green.	Pale clear blue.	Clear bluish green.
Very slight trace.	Slight trace.	Moderate trace.	Slight trace.	Moderate trace.	Slight trace.	Slight trace.	Heavy trace.	Heavy trace.	Very heavy trace.	Large amount.	Moderate trace.	Slight trace.
None.	None.	None.	None.	None.	None.	None.	None.	None.	None.	None.	None.	None.
Moderate blackening and slight fusing.	Moderate blackening and slight fusing.	Very black, and fuses.	Slightly blackens and fuses.	Slightly blackens and slight fusing.	Slightly blackens and much fusing.	Moderately blackens, and slight fusing.	Very black and fusing, bad looking.	Moderately blackens, fusing, bad looking.	Fuses, and slightly blackens, rather bad.	Moderately blackens.	Moderately blackens.	Slightly blackens.

☞ All results are given in grains per gallon, except Free and Albuminoid Ammonia, which are in parts per million.

number of bacteria it contained, it was necessary to be very careful, although, as he had said, if a large number of bacteria was constantly found, it might be taken that the water was bad. The character of the bacteria, however, was of the greatest importance. No doubt waters from different sources had their own special bacterial flora, but certain bacteria might enter into water from animal excreta, these bacteria mostly belonging to the group of organisms known as *Bacillus coli communis*. These bacilli were found in large numbers in the intestinal contents of almost all animals, and they could be distinguished from somewhat similar bacteria which were present in pure water. The bacteria present in pure water which resembled coli were bacteria belonging to the *Aquatilis sulcatus* group. They could be distinguished quite readily from coli bacilli, among other points, by the fact that they did not grow at the temperature of the body, or only grew very feebly at that temperature. The coli bacilli, on the other hand, grew readily at this temperature.

The intestinal contents of animals, human or otherwise, contained a large number of different varieties of coli, some coagulating milk quickly and some slowly, some producing more acid than others, and so on. In fact, in all intestinal evacuations, there were to be found what were called *a*-typical coli bacilli, which differed from the ordinary text-book description in some minor points. There was no evidence that coli ever occurred in water that had not been contaminated with animal matter. Water contaminated with vegetable matter and upland waters did not contain coli, as far as was known. Whenever the coli bacillus was found in any numbers, it meant contamination with animal excreta. In saying that water contained coli bacilli, he meant in fairly large numbers, since animal excremental matter was very widely distributed, and consequently coli bacilli were also widely distributed; so that in examining a large quantity of water, one might now and then meet with one or two colonies, the occurrence of which would not be of any significance, but due merely to accidental causes. A large number, however, say 60 per c.c., would definitely show a water to be contaminated with animal excreta, such as he had found to be the case in one of the springs which supplied the Farleigh water. The presence of these coli bacilli simply meant that the water was contaminated with animal excreta. It did not necessarily indicate human excreta, but it did indicate that the water was contaminated with animal excreta, and that it was thus liable to contamination with typhoid material. There was one other point with regard to the coli bacillus, namely, the question of its pathogenic properties. There was no function possessed by the coli bacillus that varied to a greater extent than its pathogenicity. Some coli bacilli had strong pathogenic properties, while others had no pathogenic properties whatever, so that this factor was of no value at all in determining whether any particular organism belonged to the coli group or not. The only point on which he disagreed with Dr. Adams was in regard to the wave of diarrhœa which preceded the typhoid epidemic. He had no doubt whatever that Dr. Adams's explanation of the rise in the subsoil water was correct, and that the water had become contaminated with typhoid virus in the way which Dr. Adams had described, and he (Dr. Washbourn) believed that the water at the same time became contaminated with the virus that produced the premonitory diarrhœa. But he believed that this virus was a living micro-organism and not a chemical poison. A large quantity of a toxalbumin would need

to be present in order to produce so much diarrhœa, and he did not know of any toxalbumin that would produce such an effect in small quantities. The more one knew about cases of "food poisoning" the more one was inclined to look upon them as due to the ingestion of bacteria, and not of toxines. He believed that the Farleigh water was contaminated both with the specific typhoid bacillus and with some form of bacterium which would produce diarrhœa, and that the reason why the diarrhœa occurred earlier was simply that the incubation period of the diarrhœa was shorter than that of typhoid fever.

Dr. CHILDS, rising in response to an invitation from the President, said that the subject of subsoil water was one of which he had made a special study in connection with the prevalence of typhoid fever in the city of Munich during the last forty years. It would be difficult, however, to make any comparisons between the data that came from Munich and those obtained from a study of the epidemic at Maidstone, though there was this analogy between them, that in Munich, as well as at Maidstone, the relations between the movements of the subsoil water and the rainfall seemed to be exceedingly irregular. It could not be predicted, from a previous heavy rainfall, whether there would be a large rise in the subsoil water or not. It depended upon a good many other conditions, such as those which had been mentioned by Dr. Adams, and also upon the previous rainfall. If the subsoil had become water-logged, the rain falling upon it would cause a considerable rise; but if there had been a drought, the subsoil itself would act as a sponge, and there might be little or no rise. The whole question required a tremendous amount of elaboration and study before one could speak of these relations with any accuracy. With regard to the relations of the subsoil-water level to typhoid epidemics, these had been very distinctly established in Munich, whilst, contrary to the experience in our own country, the Munich authorities considered that there was no relation at all between the actual drinking of the water and the prevalence of typhoid fever. Pettenkofer and his school had long been convinced of this, and, as time went on, seemed to become even more convinced still. But the conditions of the city of Munich were exceptional, and different from those usually prevailing in this country. And although the revelations obtained there were rather extraordinary, and tended to shake one's faith in the convection of typhoid by water, they could not be taken as analogous to the results generally obtained in this country. His own personal conclusion was that there were many different ways in which typhoid fever might be conveyed. The evidence obtained in this country as to the water convection of typhoid was so strong, and reached such high degrees of probability in many instances, that he thought it must be held to be conclusive, in spite of the different ideas held by such a great authority as Pettenkofer. He agreed with the President in feeling grateful to Dr. Adams for emphasizing the necessity for the establishment of a local standard of purity, and for proper inspection and supervision of the sources of water-supply and their surroundings; which latter point he (Dr. Childs) regarded as of primary importance.

Mr. HEHNER said he was sure that Dr. Adams would have the Society's sympathy in the fact that, after so many years of service at Maidstone, he should have been confronted with the recent typhoid epidemic, coming as it did after many years of labour in the interest of the health of the district. On the other hand,

Dr. Adams must be congratulated on the exceedingly interesting observations which he had made on the subsoil water of the district, which, though not appearing at first likely to lead to any useful result, had now been rewarded by the brilliant deductions he had been able to make with their aid. The long series of analyses of samples of water showed conclusively that, with one exception, they were all polluted to a large extent. Anybody conversant with water analysis, and anyone who had urged, as he (Mr. Hehner) had done many years ago, the importance of not judging from arbitrary standards, but taking as a basis the composition of the pure water of the district, would agree that there was overpowering and conclusive evidence in these analyses that the samples were polluted with a nitrogenous substance which yielded nitric acid, and hence almost certainly with animal matter. With this was to be compared the fact that even in the able hands of Dr. Washbourn bacteriology had failed in almost every case to afford evidence of pollution. He (Mr. Hehner) admitted, of course, the great difficulty of the task which had been given to Dr. Washbourn, who had had to examine the water so long after the outbreak; but Dr. Adams had also been in the same position. He (Mr. Hehner) was puzzled by some of the figures given by Dr. Adams, because, if the history of the outbreak as gathered from the newspapers was correct, the pollution was due to the accidental contamination of the soil by a limited number of people, and it was difficult to understand how on so many days, extending over some months, there should have occurred such a notable increase in the nitric acid, an increase which was found, not in one sample only, but throughout the series. The Farleigh water was supplied to 20,000 inhabitants, the average supply being about 20 gallons a day per head; consequently an increase of 110 per cent. of the normal proportion of nitric acid meant a daily increase of 126 lb. of nitric acid, which over several months would represent a good many tons, and it was scarcely possible to think that a chance pollution on the surface should have been able to produce such immense chemical results. Dr. Adams's very ingenious distinction between toxalbumin poisoning and actual bacterial poisoning struck him as somewhat bold, because there appeared to be no basis for such a theory, except the inference that premonitory diarrhœa occurred immediately before the epidemic, and before the bacilli were likely to act, a suggestion, however, which was not entertained by Dr. Washbourn, who thought that even the poisoning usually attributed to ptomaines in tinned goods was due to bacterial causes. There was, however, a sharp, or at any rate a fairly sharp, distinction between strictly chemical poisoning and bacterial poisoning. The effect of chemical poisoning was far more rapid than would be expected, if bacteria had first to incubate and multiply. But as a matter of fact the products of living organisms were capable of producing powerful toxicological effects. It might be quite possible for even a very small quantity of a substance of the nature of toxalbumin to produce the diarrhœa which had been observed.

Mr. ALLEN said that Dr. Adams had shown very clearly the great value to be attached to a conclusion derived from the quantity of oxidized nitrogen present in water. The Maidstone epidemic had been caused unquestionably by water that did not contain more than the normal proportions of free and albuminoid ammonia, etc., but contained a large proportion of nitrates. It was a fact to be greatly deplored,

that the significance of oxidized forms of nitrogen in water was systematically ignored by the writer of one of the most popular and widely-used works on water analysis. In water analysis it was impossible to have too many data upon which to base an opinion. He would say, obtain as many chemical factors as possible, and, if bacteriology was likely to be of service, make use of it also. He would not limit the analysis of water to any small number of tests, but would have as complete an examination as possible, so as to be in a position to regard the matter from every point of view. With regard to the instance in which the Government chemists, having undertaken to report upon a sample of water from a Maidstone well which was surrounded by houses infected with typhoid fever, stated that the water contained 7 grains of nitric acid per gallon, and yet was not even *liable* to contamination, he thought it was a pity that they should have gone out of their way to express an opinion on a point wholly outside their range of information, and not warranted in any way.

Mr. CASSAL said that up to the present full chemical analysis, supplemented by microscopic examination, had been more reliable, and had given more satisfactory results in water examination than bacteriology. As an aid in forming an opinion, bacteriological investigation was occasionally valuable, but the information it gave never amounted to anything more than mere confirmatory evidence. In the Maidstone investigation, in those cases where Dr. Washbourn had been able to make a positive statement, Dr. Adams would have been able to speak most positively, and no doubt did so, even from the yield of albuminoid ammonia alone; and, of course, when the nitrates also were considered, there was unquestionable, and, indeed, crushing, evidence in support of the position which Dr. Adams had taken up. There was no doubt that the detection of a large number of coli bacilli, or the detection of a large number of bacteria of any kind over and above what was normally found in a given supply, afforded valuable confirmatory evidence; but that was all, up to the present, that bacteriology had been able to do. He should like to point out that for a considerable period Dr. Adams had examined the water of his district. By this examination he was enabled to obtain what he described as his local standard, and was thereby able to gauge with accuracy any change that might take place in the character of the supply; but on the ground of so-called economy the local authority decided to dispense with this measure of precaution. They and the population they were supposed to represent had been punished most severely for that action, and the lesson was one which ought not to be forgotten.

Mr. F. WALLIS STODDART said he had been reluctantly forced to the conclusion that the detection of the typhoid bacillus under the usual circumstances of an epidemic was impracticable. He would go even further, and say that he did not think it ought to be undertaken, except as a piece of scientific research. It was desirable that something like a definition of *Bacillus coli communis* should be formulated. He had obtained pure cultures from the original discoverer, Escherich, but the organism obtained from these pure cultures was quite different from anything that had been obtained by himself, or by any of his bacteriological friends, and as long as that state of affairs existed, he felt some diffidence in saying that any organism was *Bacillus coli*, or that it was derived from intestinal sources. He was obliged to

support Mr. Hehner and Mr. Cassal in saying that unquestionably, as a guide to the quality of ground waters, there was nothing to compare with chemical analysis judiciously applied. He thought the relation of nitrates to the organic matter of sewage, both dead and living, was, perhaps, scarcely appreciated. The formation of nitrates was the outcome of a series of fermentations of dead organic matter, and it had been experimentally demonstrated that the process of nitrification could be carried out to completion without apparently interfering with the vitality of any pathogenic organisms that might be present. He gathered that Dr. Adams shared the current opinion that water falling on the surface of the ground actually sank uniformly, so to speak, through the soil, and underwent a gradual process of filtration, which resulted, first, in the elimination of these organisms; secondly, in the production of nitric acid; and ultimately in the complete purification of the water. This, however, he thought was not the case. The water travelled, and was to be obtained in quantity, only in natural passages, either between impervious strata or through fissures in those strata. Even in the case of such a pervious medium as the chalk, the water did not pass uniformly through in bulk. It invariably found the easiest channel to flow through, and nitrification was all the more complete as the water became more completely aërated during its flow. The very openness of the channel would permit the organisms to travel through a considerable thickness of subsoil. From these considerations he thought it quite possible that a water like many of these samples might be quite devoid of chemically demonstrable organic matter, and yet contain pathogenic organisms in an active and vigorous condition.

Dr. VOELCKER, while agreeing as to the desirability of local standards, said that it appeared to him to be a not very easy thing to fix a proper local standard. If the standard taken was that of a doubtful source, wrong conclusions would necessarily be formed. The real difficulty was to know whether the local standards adopted were really representative ones or not. Mr. Cassal had remarked that some of Dr. Adams' samples might have been condemned on account of the albuminoid ammonia alone. He (Dr. Voelcker) would have been very sorry to do that in any case without knowing absolutely the nature of the supply. Nor could he altogether subscribe to Dr. Adams's theory of zones. The nature of the soil seemed to have been left very much out of account. It would hardly be possible to divide all soils up into zones, and to assume that a certain set of operations went on in one zone and not in another, independently of the nature of the soil.

Dr. ADAMS, referring to Dr. Washbourn's remarks with reference to the probable cause of the premonitory diarrhœa that had occurred, observed that this diarrhœa occurred at the very commencement of the epidemic, and before the typhoid really set in, and ceased absolutely immediately the typhoid began. The two disorders must be totally and entirely different. The one exhibited as clearly as possible the characteristics of a chemical poisoning, and the other those of a bacterial poisoning. The typhoid continued to spread over a considerable time, as long, in fact, as the water containing the organisms was allowed to be drunk; the diarrhœa, on the other hand, without any interposition, occurred at the commencement of August, and concluded practically within that month, whereas the water which was believed to have been the cause was being freely drunk until the 26th or 28th of the following

month. What he maintained was, that during the whole of the dry season the putrefactive changes in the animal organic matter were going on, and that these changes brought with them certain chemical results, which accumulated on the surface, and which, being soluble, were at once carried into the water when the rain fell, and were thus consumed. Their effect then ceased, because there was no continuance of the causes which produced, and therefore no reproduction of the poisonous material. If the diarrhœa had been caused by bacteria, which simply differed from the typhoid organism in having a shorter incubation period, it might reasonably have been expected that such diarrhœa organisms would multiply and continue their effect in like manner with the typhoid organisms; but that was not so: the diarrhœa began and was ended before the typhoid started. With regard to Mr. Stoddart's observations, he of course had in view the question of local circumstances, which must be taken into consideration in the selection of local standards; though he might remark that all the waters to which he had referred in the paper came from soils which, for all practical purposes, might be regarded as precisely similar.

ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

FOODS AND DRUGS ANALYSIS.

Detection of Citric Acid in Vegetable Juices, Wine, and Milk. G. Deniges. (*Rev. Chim. Analyt. appl.*, vol. vi. [7], pp. 110-112.)—The method is based on the fact that under the influence of manganic oxidizing agents, and in an acid medium, citric acid forms with mercury a compound insoluble in presence of mercuric sulphate. It is applied as follows:

(*a*) *Free or Combined Citric Acid in Aqueous Solution.*—Five c.c. of a 1 to 2 per cent. citric acid solution are heated to boiling along with 1 c.c. of mercuric sulphate —prepared from mercuric oxide, 5 grammes; concentrated sulphuric acid, 20 c.c.; water, 100 c.c.—and, on removing from the flame, are treated with 5 to 6 drops of a 2 per cent. solution of potassium permanganate—or 1 drop if the liquid be very dilute —which decolorizes the liquid and throws down a white precipitate. This test will reveal the presence of 0·5 milligramme of citric acid.

(*b*) *In Vegetable Juices.*—A few drops of the juice are diluted with 4 to 5 c.c. of water and tested as above.

(*c*) *Small Quantities of Citric Acid in Presence of a Large Amount of Tartaric Acid.* —Five c.c. of a 2 per cent. solution of the average sample are heated with 1 c.c. of 2 per cent. permanganate until the mixture acquires a brown tinge and disengages a few bubbles of gas. When decoloration has set in, 1 c.c. of mercuric sulphate solution is added, and the whole is again heated to boiling. A decided white turbidity is obtained in presence of less than ½ per cent. of citric acid.

(*d*) *In Wine.*—Ten c.c. of wine are well shaken up with 1 to 1½ grammes of lead

dioxide, followed by 2 c.c. of mercuric sulphate solution. After filtration, 5 to 6 c.c. are heated to boiling and treated with 1 drop of permanganate, 9 additional drops being added in succession as decolorization ensues.

Under this treatment normal wines give merely a very slight haze, not always appearing at once, due to the traces (5 to 6 centigrammes per litre) of citric acid invariably present, a fact not hitherto recorded. With 0·1 gramme per litre the turbidity is pronounced, and above 0·4 gramme a flocculent precipitate is formed.

(*e*) *In Milk.*—Ten c.c. of milk, 2 c.c. of a 5 per cent. solution of sodium metaphosphate, and 3 c.c. of the above mercuric reagent, are shaken up together and filtered, the first runnings being rejected, 5 to 6 c.c. heated to boiling and shaken up with successive drops of the permanganate solution. A decided white turbidity ensues after the addition of 4 or 5 drops in the case of cows' milk, and with 8 to 10 drops a flocculent white precipitate accompanied by a yellow coloration, removable by hydrogen peroxide.

The author has ascertained that acetic, tartaric (etc.) acids, glycerin, gum, and other substances likely to occur with citric acid, do not interfere with the reaction; any excess, however, of chlorides, bromides, or iodides, must be first eliminated by silver sulphate. When oxalic acid or other substance capable of reacting on mercuric sulphate is present, the mixture must be first oxidized in an acetic medium by a slight excess of permanganate, which is then reduced by hydrogen peroxide before proceeding to apply the test. C. S.

Adulteration of Pimento. T. F. Hanausek. (*Zeit. für Untersuch. der Nahr. und Genussmittel*, 1898, 245.)—The author calls attention to the adulteration of pimento with roasted cacao husks. The adulteration may be known by the presence of homogeneous acute-angled particles of a reddish-brown colour bordered by dark brown on one side or on two opposite sides. Treatment with hot water or solution of potash yields a slimy membrane, and particles may be seen composed of a spongy tissue with sclerogen. Adhering to particles of spongy parenchyma are bundles of spires. These also occur separately, and contain spiral fibres, often in the form of open rings. H. H. B. S.

Determination of Oil of Mustard. E. Haselhoff. (*Zeit. fur Untersuch. der Nahr. und Genussmittel*, 1898, 235.)—The methods for the determination of oil of mustard depend upon the estimation of the sulphur or nitrogen contained in it.

Förster (*Landw. Versuchs. Stat.*, xxxv., 209) converts the oil into thiosinamin, and precipitates the sulphur with mercuric oxide as mercuric sulphide.

Schlicht (*Zeit. anal. Chemie*, xxx., 661) distils with an alkaline solution of potassium permanganate, reduces the excess of permanganate with alcohol, and determines the sulphuric acid by barium chloride.

Passon (*Zeit. angw. Chemie*, 1896, 422) determines the nitrogen by Kjeldahl's method.

The author has made a comparative examination of these three methods as well

as of a modification of Schlicht's method, consisting in the use of bromine-water in place of potassium permanganate. The mean results are given in the following table:

Amount present.	Found by Förster's Method.	Found by Schlicht's Method.		Found by Passon's Method.
		Permanganate.	Bromine.	
.96·72	95·34	95·44	96·47	95·44

According to Schuster and Mecke (*Chem. Zeit.*, xvi., 1954), the rise in temperature which takes place on milling rape-seed prior to pressing out the oil increases the percentage of oil of mustard in the seed threefold. The author's experiments do not confirm this. In the following case a small increase resulted on heating to 70° C., but by no means to the extent mentioned by Schuster and Mecke:

Fresh rape-seed	0·305	per cent.	oil of	mustard.
Defatted rape-seed	0·290	,,	,,	,,
Rape-seed heated to 70° C	0·347	,,	,,	,,

H. H. B. S.

INORGANIC ANALYSIS.

Volumetric Determination of Antimony. G. Rollin. (*Rev. de Chim. et de Pharm.*, 1898, February 10, p. 129; through *Rev. Chim. Analyt. appl.*, vol. vi. [7], pp. 114, 115.)—The author bases his method, which is applicable to the sulphides and commercial oxides of the metal, on the following considerations:

1. The Mohr titration is accurate provided the solution is kept alkaline by means of a bicarbonate throughout the operation.

2. Arsenic sulphide is completely insoluble, whereas the antimony salt is soluble in *cold* concentrated hydrochloric acid, and so long as the substance does not contain more than 5 parts of arsenic oxide per 100 of antimony oxide, the precipitate produced under these conditions is free from the latter metal.

3. A current of air has no action on cold solutions of antimony chloride.

4. A solution of antimony in a large excess of hydrochloric acid may be boiled for a long time without loss of metal.

In testing commercial oxides of antimony, 1 gramme of substance is dissolved in 10 c.c. of hydrochloric acid of a specific gravity of 1·176, and treated with gaseous sulphuretted hydrogen to remove arsenic. The liquid being then transferred to a 250 c.c. flask and the glass rinsed with a little hydrochloric acid diluted with its own bulk of water (not more), the excess of sulphuretted hydrogen is removed by a current of air. Five grammes of tartaric acid are added, the liquid made up to the mark, and passed through a dry filter. One-tenth of this volume is taken for titration, carefully saturated with sodium bicarbonate solution, and, after adding a pinch of the solid salt and a little starch paste, is titrated with standardized iodine solution.

In the case of antimony sulphide, 1·5 grammes are dissolved in hot concentrated

hydrochloric acid, and, when *perfectly cold*, treated by sulphuretted hydrogen, etc., as before. C. S.

Separation of Thorium from Cerite Earths. Wyrouboff and Verneuil. (*Comptes rendus*, vol. cxxvi., p. 340; through *Rev. Chim. Analyt. appl.*, vol. vi. [7], pp. 112, 113.)—The method is based on the transformation of thoria into peroxide by means of hydrogen peroxide (proposed by Clève). The substance is dissolved in nitric acid, and a quantity of the solution corresponding to 0·5 gramme (maximum) of the oxides is evaporated to dryness, and treated with 100 c.c. of water and an equal quantity of hydrogen peroxide, the whole being heated and stirred for several minutes. The bulky gelatinous precipitate is washed until the washings no longer give a precipitate with ammonia, and, after removal from the filter, is redissolved in a little hot water containing 2 grammes of ammonium iodide and 2 c.c. of concentrated hydrochloric acid, the hot solution being passed through the filter to dissolve any residual peroxide thereon. The hydroxide thrown down by ammonia is thrown on the same filter and calcined without washing, the filtrate being treated with ammonia to precipitate the other earths present in the substance.

If the precipitate produced by hydrogen peroxide is not perfectly white, it contains cerium, and should be purified by re-solution in nitric acid and treating as before—a course advisable in any case, to ensure at the same time the total precipitation of the thorium present. C. S.

Differentiation of Alkali Bicarbonates and Carbonates in Mixtures. A. Leys. (*Ann. Chim. Analyt.*, vol. iii. [2], pp. 44-46.)—The precipitation of neutral alkali carbonates by magnesium salts being masked by the solvent action of an alkali bicarbonate or of borax in mixtures, the author has studied the reaction of the calcium salts in this connection, and finds that by using a saturated solution of calcium sulphate the neutral carbonates are thrown down immediately as an opaque white precipitate, whilst precipitation of the bicarbonates occurs only after prolonged contact with this precipitate. The crystalline powder, which then separates out, is readily distinguishable from the former. Thus, in the case of "pure" commercial sodium bicarbonate, if a quantity of water insufficient for complete solution be added, and a portion of the solution dropped into the reagent, an opaque precipitate indicating neutral carbonate is obtained. C. S.

Separation and Estimation of Iodine, Bromine, and Chlorine. A. Carnot. (*Comptes rendus*, vol. cxxvi. [3], p. 187; through *Rev. Chim. Analyt. appl.*, vol. vi. [3], pp. 34-36.)

Iodine.—The neutral solution of chlorides, bromides, and iodides is diluted to 200 c.c. and placed in a stoppered 400 c.c. flask, terminating below in a narrow tapped tube. Here the iodine is liberated by means of 10 drops of sulphuric acid saturated with nitrous fumes, and extracted by shaking the liquid up with 10 to 15 c.c. of carbon disulphide, which, on settling, is drawn off through the tap.

This operation is repeated twice, and on the last occasion the solvent should remain colorless. The carbon disulphide is placed on a moistened filter and the first washings are returned to the original liquid, the disulphide being then run through the perforated filter into a flask, where it is mixed with 30 c.c. of a ½ per cent. solution of sodium bicarbonate and titrated with standard sodium thiosulphate.

Bromine.—Several c.c. of 10 per cent. chromic acid and 3 to 4 c.c. of sulphuric acid diluted with its own volume of water are added to the liquid separated from the iodine, and the closed flask is placed in a boiling-water bath for an hour. The bromine is then extracted from the cooled liquid by carbon disulphide as in the case of the iodine, the disulphide solution being well shaken up with a little potassium iodide and 30 c.c. of ½ per cent. sodium bicarbonate, and the liberated iodine titrated with sodium thiosulphate; the bromine is found by multiplying the weight of iodine by 0·6308.

Chlorine.—The residual acid solution is diluted to 500 c.c., and warmed gently along with silver nitrate to cause the precipitate to collect. The latter is purified from the silver chromate usually present by treatment with hot water slightly acidified with nitric acid, and is finally washed, dried, and weighed. C. S.

Determination of the Strength of Hydrofluoric Acid Solutions. J. Zellner. (*Monatsheft. für Chemie*, xviii., 749.)—A volumetric process is recommended, consisting of titration by potash, using phenolphthalein as the indicator. A moderate excess of alkali is added, the solution boiled for a short time, and titrated back while hot. Comparative tests made volumetrically, in this way, and afterwards in the same portion gravimetrically, gave the following results, in parts per cent.:

	I.	II.	III.	IV.
By volumetric method	5·22	7·63	21·27	31·40
By gravimetric method	5·20	7·68	21·56	31·65

If the titration is carried out in the cold, the results, though agreeing between themselves, are about 1 per cent. below the gravimetric tests. For weighing the quantity of hydrofluoric acid taken for the determination, a cylindrical vessel of hard indiarubber is used, having an outlet at the bottom through a flexible small bore caoutchouc tube provided with a pinchcock. The apparatus, which weighs about 60 grammes, can be secured to the pan of the balance by a piece of platinum wire. It is first weighed full, and then after running out the required quantity of acid, the weight taken being obtained by difference. H. H. B. S.

The Determination of Boric Acid as Potassium Borofluoride. K. Thaddéeff. (*Zeit. anal. Chem.*, 1897, xxxvi., 568-637.)—After giving a summary of the various methods employed for the determination of boric acid, and showing the unreliability of any of those based on an estimation by difference, the author describes a modification of the Berzelius-Stromeyer method which he considers highly satisfactory when carried out exactly as described below. When in combination with a base, except potassium, the boric acid is distilled over with sulphuric acid and methyl alcohol, as in Rosenbladt's apparatus, a regulated air-current being at the same time introduced into the distilling flask. The distillate from the substance containing

1 gramme or less of boric acid is received in a platinum basin containing a 10 per cent. solution of pure caustic potash, and when four successive quantities of 10 c.c. of methyl alcohol have been distilled over, is concentrated to half its volume on the water-bath. An excess of pure hydrofluoric acid is then added, and the evaporation continued until only a faint smell of hydrofluoric acid is perceptible. When cool, 50 c.c. of a solution of potassium acetate (specific gravity 1·14) are added, and the basin is allowed to stand for one or two hours at the ordinary temperature, the mass being frequently stirred with a platinum rod so as to ensure the whole of the potassium hydrogen fluoride dissolving. 100 c.c. of alcohol (specific gravity 0·805) are next added, the liquid carefully stirred and allowed to remain over-night.

Meanwhile, a filter-paper has been prepared (a No. 590 Schleicher and Schüll, 9 c.m. in diameter), moistened with alcohol, dried at 100° to 110° C., and weighed in a stoppered weighing-flask. The insoluble residue of potassium borofluoride is washed on to this filter with alcohol of 0·805 specific gravity, and the washing continued until on evaporating a few drops of the filtrate on platinum foil, no more residue remains than the small amount of borofluoride soluble in the alcohol (0·000014 gramme per c.c.). From 62 to 72 c.c. of alcohol are usually required. The filter and precipitate are then dried at 100° to 110° C. for three hours, and weighed. The following are some of the results obtained in this way with borax:

Borax taken. Grammes.	KBF_4. Grammes.	B_2O_3 formed. Grammes.	Per cent.	Difference from theory. Per cent.
1	1·3229	0·3653	36·53	−0·06
0·5	0·6631	0·1831	36·62	+0·03
0·1	0·1308	0·0361	36·12	−0·47

C. A. M.

The Commercial Analysis of Bauxite. W. B. Phillips and D. Hancock. (*Jour. Amer. Chem. Soc.*, 1898, xx., 209-225.)—The author states that the formula given by Roscoe for bauxite, $(AlFe)_2O(OH)_4$, does not apply to the American mineral, which consists essentially of a mixture of aluminium trihydrate with clay and another aluminium compound, possibly a lower hydrate.

In assaying the material for alum manufacture it is usual to evaporate it with sulphuric acid until the latter begins to fume; but in this way aluminium compounds are brought into solution which are not very suitable for the manufacture, and are certainly of very much less value than the readily soluble trihydrate. The author therefore recommends that the alumina soluble during one hour in sulphuric acid of a specific gravity of 1·516, at 100° C. shall be described as "free alumina," and that soluble on evaporating the acid to fumes as "available alumina," whilst the difference between the two shall be known as "combined alumina." He describes experiments which prove that practically the whole of the aluminium trihydrate is dissolved by the first treatment, and that concordant results can be obtained.

The analytical method recommended is as follows: The sample is finely powdered and passed through a sieve of 100 meshes to the inch.

Moisture.—Two grammes are dried to constant weight at 100° C.

Available Alumina.—Two grammes of the bauxite are mixed with 10 c.c. of cold

sulphuric acid (S.G. 1·516), and the basin gradually heated until fumes begin to appear. It is then covered with a glass, and the heating continued for ten minutes. When cool, 100 c.c. of hot water are added, and after being boiled for five minutes the liquid is filtered, the residue washed, and the filtrate and washings made up to 200 c.c.

Fifty c.c. are diluted to 300 c.c., 2 c.c. of hydrochloric acid added, the liquid boiled, ammonia added in slight excess, and the boiling continued for five minutes. The precipitate is filtered off, washed, dried, ignited, and weighed as alumina, ferric oxide, and titanium dioxide, and the two latter are separately determined and deducted.

Estimation of Titanium Dioxide.—To 50 c.c. of the main solution, ammonia is added until there is a slight precipitate. This is just dissolved in sulphuric acid, and the liquid is made up to 350 to 400 c.c. and boiled for an hour. Any iron present is reduced by means of sulphur dioxide, care being taken that the liquid smells of the gas during the boiling. In this way the titanium dioxide is precipitated practically free from iron. The precipitate is filtered while hot through a double filter, washed with hot water, dried, ignited, and weighed.

Estimation of Ferric Oxide.—This is determined in the filtrate from the titanium dioxide by reduction with zinc and titration with permanganate.

Estimation of "Free Alumina."—Two grammes of bauxite are mixed with 10 c.c. of sulphuric acid (S.G. 1·516) in a 4-oz. Erlenmeyer flask, which is provided with a perforated stopper. The flask is heated on the water-bath at 95° to 100° C, with frequent shaking for an hour, after which 100 c.c. of hot water are added and the flask kept in the water for ten minutes. The liquid is filtered, the residue washed with hot water, and the alumina determined in the filtrate as before, with the exception that the titanium dioxide, which is not dissolved under these conditions, is not determined. The precipitate consists of alumina and ferric oxide, and the latter is separately determined in the usual way and deducted.

Combined Alumina is the difference between the available alumina and the free alumina.

In order to determine the silica and the total alumina, etc., the insoluble residue left on treating the mineral with the sulphuric acid may be ignited with the filter-paper, the ash fused with potassium bisulphate, and the melt left in cold water containing at least 5 per cent. of sulphuric acid until decomposed. The silica is filtered off, ignited, and weighed, and the alumina, ferric oxide, and titanium dioxide determined in the filtrate, and added to the amounts found in the determination of the available alumina.

It is not advisable to determine the silica in the insoluble residue by treatment with hydrofluoric acid, since some of the titanium dioxide is simultaneously volatilized.

C. A. M.

NOTE.—It is well known that if sulphuric acid, as well as hydrofluoric, is employed in the elimination, the titanium dioxide is completely retained.—B. B.

Analysis of Calcium Carbide. H. Bamberger. (*Zeit. angew. Chem.*, 1898, 196-198.)—In place of the ordinary methods of determining volumetrically the amount of acetylene obtainable from a given weight of carbide, which have the draw-

back that a correction must be made for the gas absorbed by the liquids with which it comes in contact, as well as the ordinary corrections for temperature and pressure, the author prefers to use a gravimetric method; the principle of which is the same as that for the determination of carbonic acid in carbonates by difference.

C. A. M.

On the Products of the Decomposition of Calcium Carbide by Water. E. Chuard. (*Bull. Soc. Chim.*, 1897, xvii., 678, 679.)—The author has established the presence of ammonia not only in the acetylene liberated from the carbide, but also in the residue. For 100 parts of calcium carbide the gas contains from 0·03 to 0·06 of ammonia, the residue 0·24 to 0·40. The ammonia liberated with the acetylene is derived from the decomposition of calcium nitride, $Ca_3N_2 + 3H_2O = 3CaO + 2NH_3$, whilst that in the residue is a product of the decomposition of calcium cyanate :—

$$Ca(CNO)_2 + 3H_2O = CaCO_3 + CO_2 + 2NH_3.$$

Other impurities invariably present are phosphoretted and sulphuretted hydrogen. It is to the presence of the former, the amount of which is usually 0·018 to 0·032 per cent. on the carbide, that the insecticidal properties of crude acetylene are probably due.

C. A. M.

LEGAL.

QUEEN'S BENCH DIVISION.

COURT FOR THE CONSIDERATION OF CROWN CASES RESERVED.

Reprinted from the "Times" of May 2, 1898.

(Before the LORD CHIEF JUSTICE OF ENGLAND, MR. JUSTICE DAY, MR. JUSTICE WILLS, MR. JUSTICE GRANTHAM, MR. JUSTICE WRIGHT, MR. JUSTICE KENNEDY and MR. JUSTICE CHANNELL).

PETCHLEY *v.* TAYLOR.

THIS was a case stated by a Metropolitan police magistrate upon convicting the appellant upon an information preferred under the Sale of Food and Drugs Act, 1875, for unlawfully selling to the prejudice of the purchaser milk which had 97 per cent. of the original fat abstracted, so as to affect injuriously its quality and substance, without making disclosure of the said alteration, contrary to Section 9 of the above Act.

Mr. George Elliott appeared for the appellant; and Mr. Courthope Munroe for the respondent.

On November 13, 1897, the appellant sold to the respondent a tin containing a substance described as "Cup Brand Condensed Milk." On the tin were the words, "This tin contains skimmed milk with nothing added but the finest sugar." The substance in the tin was proved to be separated milk, or milk from which the cream had been separated by means of a machine called a separator, and that 97 per cent. of the original fat had been abstracted. It was also proved that the term "skimmed milk" meant milk from which a portion of the fat had been removed by the process of skimming the surface of the milk, and that the greatest amount of fat that could be thus removed was 63 per cent.

Mr. Elliott contended that the alteration was sufficiently disclosed by the term "skimmed milk," and cited "Jones *v.* Davies" (69 L.T., 497) and "Platt *v.* Tyler" (58 J.P., 72).

The Court yesterday dismissed the appeal.

THE ANALYST.

JULY, 1898.

PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS.

THE monthly meeting of the Society was held on Wednesday evening, June 1, in the Chemical Society's Rooms, Burlington House, the President (Dr. Bernard Dyer) occupying the chair.

The minutes of the previous meeting were read and confirmed.

Mr. H. J. Lewin was elected a member, and Mr. F. M. Wharton an associate of the Society.

Mr. G. W. Slatter, A.R.C.S., F.I.C., was proposed for election as a member, and Mr. A. J. Bull, assistant to Mr. Chattaway, was proposed for election as an associate.

The PRESIDENT, referring to the recent death of Lord Playfair, who had been an honorary member of the Society almost from the beginning of its history, moved from the chair that a message of condolence be forwarded to Lady Playfair by the Society. The motion was carried unanimously.

The following papers were read:

"Note on some Apparatus." By H. Droop Richmond.

"Sewage Analysis and Standards of Purity for Effluents." By C. G. Moor, M.A.

"Note on Certain Resins." By Rowland Williams.

"The Proportion of Oxygen Present in Linseed-Oil, both before and after Oxidation." By Rowland Williams.

"Variations in the Composition of Chicory." By Bernard Dyer, D.Sc.

"The Sulphuric Acid in Portland Cement." By Eug. Ackermann.

THE CALCULATION OF "ADDED WATER" IN ADULTERATED MILKS.

By H. DROOP RICHMOND.

(*Read at the Meeting, March* 16, 1898.)

IF we add to or take from a milk any quantity of fat, we shall not affect the percentage of solids-not-fat in the "milk devoid of fat."

Calling the solids-not-fat S, and the fat F, the following equation holds true for any given milk, and the cream and skim milk prepared from it:

$$\frac{S}{100-F} \times 100 = K_1 \ldots\ldots\ldots (1).$$

K_1 represents the percentage of solids-not-fat in the "milk devoid of fat."

I have already shown that the following equation connects total solids, fat, and specific gravity (ANALYST, xix., 81):

$$T = 0{\cdot}2625\,\frac{G}{D} + 1{\cdot}2\,F \ldots\ldots (2);$$

where T = total solids;
G = degrees of gravity (water at 60° = 1000);
D = density (water at 60° = 1);
F = Fat.

[D and G are different modes of expression of the same fact; the expression $\frac{G}{D}$ can be expressed either in terms of G, as $\frac{1000\,G}{1000+G}$, or in terms of D, as $\frac{1000\,D-1000}{D}$; the expression $\frac{G}{D}$ is adopted as being less cumbersome. It really represents "degrees of specific volume."]

I have also shown (ANALYST, xx., 57) that equation (2) may be written:

$$T = 0{\cdot}25\,G + 1{\cdot}2\,F + 0{\cdot}14 \ldots\ldots (3).$$

The difference between the results given by equations (2) and (3) does not exceed 0·02 per cent.

It is evident that as the total solids are composed of fat and solids-not-fat, we can substitute F + S for T in equation (3), which is then written:

$$F + S = 0{\cdot}25\,G + 1{\cdot}2\,F + 0{\cdot}14,$$

or,
$$S = 0{\cdot}25\,G + 0{\cdot}2\,F + 0{\cdot}14 \ldots\ldots (4).$$

We may substitute for S in equation (1) the value given by equation (4):

$$\frac{0{\cdot}25\,G + 0{\cdot}2\,F + 0{\cdot}14}{100 - F} \times 100 = K_1.$$

This may be written:

$$\frac{0{\cdot}25\,G + 0{\cdot}25\,F + 0{\cdot}14 - {\cdot}05\,F}{100 - F} \times 100 = K_1;$$

or,
$$0{\cdot}25\,G + 0{\cdot}25\,F + 0{\cdot}14 - 0{\cdot}05\,F = K_1 \times \frac{100 - F}{100};$$

or,
$$0{\cdot}25\,G + 0{\cdot}25\,F = K_1 \times \frac{100 - F}{100} - 0{\cdot}14 + 0{\cdot}05\,F;$$

or,
$$G + F = 4 \times K_1 \times \frac{100 - F}{100} - 0{\cdot}56 + 0{\cdot}20\,F;$$

or,
$$G + F = 4 \times K_1 - 0{\cdot}56 + 0{\cdot}20\,F - \frac{4 \times K_1}{100} \times F \ldots\ldots (5).$$

The average value of K_1, the percentage of solids-not-fat in the "milk devoid of fat," is about 9·0, and the value of $\frac{4 \times K_1}{100}$ may be taken as 0·36.

Equation (5) may be written:

$$G + F = 4 \times K_1 - 0{\cdot}56 + 0{\cdot}20\,F - 0{\cdot}36\,F;$$

or,
$$G + F = 4 \times K_1 - 0{\cdot}56 - 0{\cdot}16\,F \ldots\ldots (6).$$

For comparatively small values of F the expression $(-0{\cdot}56 - 0{\cdot}16\,F)$ will approximate to a constant, and equation (6) becomes:

$$G + F = \text{constant} \ldots\ldots (7).$$

In the following table I have given a series of analyses of milks which were known to be not watered; the samples connected by brackets were identical, but the analyses were performed by different analysts working independently:

G.	T. S.	F.	S.-not-F.	G+F.
32·0	12·55	3·79	8·76	35·79
31·8	12·44	3·70	8·74	35·50
31·2	12·27	3·65	8·62	34·85
31·9	12·38	3·67	8·71	35·57
31·8	12·24	3·50	8·74	35·30
31·2	12·09	3·58	8·51	34·78
31·2	12·36	3·80	8·56	35·00
30·2	12·38	4·08	8·30*	34·28
30·7	12·56	4·10	8·46	34·76
31·6	12·42	3·86	8·56	35·46
31·7	12·42	3·68	8·74	35·38
32·2	12·30	3·62	8·68	35·82
31·3	12·07	3·42	8·65	34·72
31·1	12·08	3·61	8·47	34·78
31·7	12·47	3·78	8·69	35·48
32·3	12·40	3·69	8·71	35·99
31·8	12·65	3·85	8·80	35·65
32·2	12·84	3·94	8·90	36·14
32·8	12·21	3·27	8·94	36·07
32·0	12·31	3·35	8·96	35·55
33·0	12·86	3·69	9·17	36·69
32·6	12·86	3·92	8·94	36·52
32·5	12·42	3·57	8·85	36·07
32·5	12·47	3·66	8·81	36·16
32·7	12·25	3·32	8·93	36·02
33·0	12·22	3·33	8·89	36·33
32·4	12·78	3·72	9·06	36·12
32·2	12·71	3·92	8·79	36·12
32·9	12·30	3·40	8·90	36·30
32·7	12·34	3·54	8·80	36·24
32·9	12·52	3·46	9·06	36·36
33·1	12·50	3·49	9·01	36·59
32·8	12·71	3·59	9·12	36·39
33·2	12·77	3·77	9·00	36·97
31·6	13·20	4·29	8·91	35·89
31·6	13·22	4·25	8·97	35·85
32·9	13·95	4·70	9·25	37·60
32·9	13·81	4·51	9·30	37·41
32·9	13·94	4·60	9·34	37·50
36·6	9·73	0·25	9·48†	36·85
36·6	9·64	0·27	9·37	36·87
36·8	9·66	0·24	9·32	37·04
33·5	12·33	3·24	9·09	36·74
33·0	12·27	3·09	9·18	36·09
33·5	12·19	3·12	9·07	36·62
32·2	12·38	3·51	8·87	35·71
32·0	12·41	3·46	8·95	35·46
32·3	12·30	3·44	8·86	35·74

* Minimum. † Maximum.

G.	T. S.	F.	S.-not-F.	G+F.
32·6	13·00	3·98	9·02	36·58
32·3	13·01	4·12	8·89	36·42
32·5	13·02	3·97	9·05	36·47
32·5	12·97	4·11	8·86	36·61
32·0	12·90	3·88	9·02	36·08
32·4	12·87	4·09	8·78	36·49
31·2	13·10	4·30	8·80	35·50
31·5	13·25	4·59	8·66	36·09
31·2	12·01	3·37	8·64	34·57
32·0	11·97	3·35	8·62	35·35
32·2	12·64	3·61	9·03	35·81
32·2	12·45	3·71	8·73	35·91
36·2	9·44	0·19	9·25	36·39
36·2	9·31	0·23	9·08	36·43
36·0	9·47	0·30	9·17	36·30
36·3	9·42	0·41	9·01	36·71
31·8	12·65	3·77	8·88	35·57
32·2	12·59	3·86	8·73	36·06
33·0	12·18	3·17	9·01	36·17
33·0	12·05	3·31	8·74	36·31
	Total mean		8·88	36·02

In these it is seen that the maximum difference between two analysts is 0·30 per cent. of solids-not-fat, or 3·3 per cent. of the mean; the maximum difference between the sums of the gravity and the fat found by two analysts is 0·78, or 2·2 per cent. of the mean. The extreme differences (excluding the separated milks) in the table amount to 1·04 per cent. solids-not-fat, or 11·7 per cent. of the mean; and of the sums of gravity and fat to 3·32, or 9·2 per cent. of the mean. It appears that the sum of the gravity and fat is not only less affected by experimental error than the solids-not-fat, but that it is a more constant figure in genuine milks.

In the following table, series of analyses of the same milk in which the fat has been varied, are given:

No.	Specific Gravity.	Total Solids.	Fat.	Solids-not-Fat.	Sum.	Deviation from Mean.
I.	1·0327	11·62	2·75	8·87	35·45	−0·10
	1·0316	12·85	4·00	8·85	35·60	+0·05
	1·0304	13·99	5·20	8·79	35·60	+0·05
II.	1·0349	9·27	0·28	8·99	35·18	+0·14
	1·0308	12·75	4·10	8·65	34·90	−0·14
III.	1·0362	9·26	0·06	9·20	36·26	−0·23
	1·0332	12·65	3·50	9·15	36·70	+0·23
IV.	1·0367	9·36	0·04	9·32	36·74	+0·02
	1·0367	9·45	0·12	9·33	36·82	+0·10
	1·0357	10·38	1·10	9·28	36·80	+0·08
	1·0330	12·59	3·50	9·09	36·50	−0·22
V.	1·0331	10·83	2·02	8·81	35·12	−0·30
	1·0315	12·40	3·65	8·75	35·15	−0·27
	1·0305	14·34	5·48	8·86	35·98	+0·56
VI.	1·0318	11·89	3·17	8·72	34·97	+0·01
	1·0312	12·45	3·75	8·70	34·95	−0·01
VII.	1·0313	12·95	4·10	8·85	35·40	−0·08
	1·0262	17·92	9·36	8·56	35·56	+0·08

It is seen from this series that the sum of the gravity and fat remains appreciably constant when considerable variations occur in the fat; the difference, except in No. V., is not greater than the divergence between the results of two analysts working independently on identical samples; No. V. is included to show the maximum difference yet observed. The table could be greatly extended, but the results given above are sufficient.

I venture to propose, therefore, that the sum of the fat and gravity be used for the calculation of added water instead of the solids-not-fat, because—

(i.) It is less affected by experimental error;

(ii.) It is a more constant figure;

(iii.) It is but slightly affected by excess or deficiency of cream.

The value 36·0 may be taken as the mean, and should be used where a mean percentage of added water is desired. From the above table, it appears that the value 34·5 may be used where a safe percentage of added water is to be certified, in the same way that the standard of 8·5 per cent. solids-not-fat is used.

In the following table some analyses of watered milks are given; the percentage of added water is calculated from the solids-not-fat using 8·88 (the mean value found above) as a standard, and from the sum of the fat and gravity using 36·0 (the mean given above):

No.	Specific Gravity.	Total Solids.	Fat.	Solids-not-Fat.	Sum.	Added Water. From Solids-not-Fat.	Added Water. From Sum.	Added Water. Actual.
1*a*.	1·0295	11·31	3·08	8·23	32·58	7·3	9·5	10
1*b*.	1·0298	11·34	3·08	8·26	32·88	7·0	8·7	10
1*c*.	1·0298	11·29	3·03	8·26	32·83	7·0	8·8	10
2*a*.	1·0243	16·11	8·18	7·93	32·48	10·7	9·8	10
2*b*.	1·0245	15·97	8·12	7·85	32·62	11·6	9·4	10
3*a*.	1·0300	10·93	2·72	8·21	32·72	7·5	9·1	9
3*b*.	1·0302	10·94	2·65	8·29	32·85	6·6	8·8	9
4.	1·0289	10·84	2·92	7·92	31·82	10·7	11·6	12
5.	1·0236	9·41	2·80	6·61	26·40	25·5	26·5	27
6.	1·0285	11·24	3·30	7·94	31·80	10·6	11·7	13
7.	1·0284	10·97	3·10	7·87	31·50	11·4	12·5	16·5
8.	1·0290	11·66	3·55	8·11	32·55	8·7	9·6	10·5
9.	1·0293	11·32	3·20	8·12	32·50	8·6	9·7	7·5

The analyses 1*a*, 1*b* and 1*c*, 2*a* and 2*b*, and 3*a* and 3*b*, were made by different analysts working on identical samples. No. 1 was milk which had been watered; No. 2 had also received an addition of cream; while No. 3 contained a proportion (30 per cent.) of separated milk. The percentages of water calculated by the method above described agree better with that actually added than the percentages deduced from the solids-not-fat.

It is not claimed that a determination of fat and specific gravity can supplant in any way estimations of total solids, solids-not-fat, milk-sugar, proteids, and ash, in judging whether a milk is genuine or not; but determinations of specific gravity and fat are always made in an ordinary analysis. It appears from the above evidence that, when watering is established by the ordinary analytical methods, a more correct estimate of the percentage of water added can usually be obtained by

calculating from the sum of specific gravity and fat; and the calculation is of the very simplest.

In this paper I have made use of analyses by Messrs. Bevan, Boseley, Harland, Hehner, and O'Shaughnessy, as well as my own, and my best thanks are due to these gentlemen for permission to use their work.

ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

FOODS AND DRUGS ANALYSIS.

The Detection of Annatto in Milk. A. Leys. (*Journ. Pharm. Chem.*, 1898, vii., 286-289.)—Fifty c.c. of the suspected milk are shaken in a separatory funnel with 100 c.c. of the following mixture made in the following proportions: Alcohol (93 per cent.), 240 c.c.; ether, 320 c.c.; water, 20 c.c.; ammonia solution (specific gravity 0·92), 8 c.c. On standing, the liquid forms two layers, the upper consisting of an ethereal solution of the fat, and the lower containing the casein, etc. The coloring matter of annatto will be found almost entirely in this lower ammoniacal layer, to which it imparts a greenish-yellow tint.

After twenty minutes the lower layer is removed to a second separatory funnel, and half its volume of a 10 per cent. solution of sodium sulphate added to it in small fractions, the funnel being inverted without shaking after each addition. The casein is thus separated in flakes, which conglomerate and rise to the surface, and the liquid is drawn off from below through wire gauze and placed in four test-tubes. To each of these amyl alcohol is added, and after shaking they are placed in a vessel of cold water, and the temperature gradually raised to 80° C., which causes the emulsion to break up, and the amyl alcohol, with the coloring matter in solution, to rise to the surface.

The residue obtained on evaporating the united amyl alcohol extracts is dissolved in warm water containing a little ammonia and alcohol. A bundle of white cotton fibre is introduced, and the solution evaporated almost to dryness on the water-bath. The yellow fibre, when washed and placed in a solution of citric or other weak acid, is instantly changed to a rose colour when the milk had been colored with annatto.

An uncoloured milk when tested in this way gives a slight yellow coloration to the cotton, but never the rose colour. Similarly negative results are obtained in the case of milk colored with saffron, turmeric, or with the coloring matter of marigold flowers, so that the reaction appears to be characteristic of annatto. C. A. M.

The Detection of Added Water in Milk. A. Villiers and M. Bertault. (*Bull. Soc. Chim.*, 1898, xix., 305-310.)—The authors state that there exists a peculiar relation between the amount of lactose and of salts in milk-whey, so much so that the refractive power of the latter is virtually a constant. They have established this in the case of thirty-eight samples of milk of undoubted purity, obtained from cows of different breed and age. In these the total solids varied from 110 to 202·4 grammes per litre; those of the whey, after boiling, from 61·20 to 74·60 grammes per litre; the soluble ash from 3·22 to 8·40; lactose from 37·80 to 58·50; and the chlorides, as sodium chloride, from 0·93 to 2·97.

The whey was prepared by adding 1 volume of 1 per cent. acetic acid to 2 volumes of the milk, bringing the mixture to the boiling-point in a flask connected with a reflux condenser, then cooling and filtering.

The total solids were determined by evaporating 5 c.c. in vacuo, and the refractive power by means of Jean and Amagat's oleorefractometer. The latter results were calculated by subtracting from the figure given by the whey diluted with acetic acid, one-third of the difference between the respective figures for the dilute acetic acid and for pure water; deducting from the result the figure corresponding to pure water, and adding to itself one half of the difference.

If, for example, 11·5 divisions were the reading in the case of pure water, 15 divisions that of the dilute acetic acid, and 41 that of the dilute whey, the result corresponding to the substances dissolved in the undiluted whey would be obtained by deducting

$$41 - \frac{15 - 11{\cdot}5}{3} - 11{\cdot}5 = 28{\cdot}3,$$

and adding half the latter number to itself = 42·4. The number of divisions corresponding to the lactose is calculated by multiplying the amount of lactose in 100 c.c. of the milk by 6·17, the number corresponding to 1 gramme of lactose in 100 c.c. Omitting samples containing colostral milk (which are easily recognised by the presence of albumin in the whey prepared in the cold), the lowest result obtained was 39·5, whilst as a rule the figure was 40 to 41. In the case of colostral milk the lowest result was 38·5.

As an example of the application of the method, two samples of genuine milk are mentioned which contained 131·20 and 110·20 grammes of total solids and 58·50 and 45·00 grammes of lactose per litre respectively, so that, even after the addition of 15 per cent. of water, the first would still give superior figures to the second in these particulars. When examined by the oleorefractometer, the first gave a reading of 41 divisions, the second 39·5, whilst the figure obtained on adding 10 per cent. of water to the first was 37.

The second sample was taken from a Dutch cow, and the authors call attention to the importance of the fact that the whey of milk obtained from Dutch cows in general is not marked by a low refractive power, notwithstanding the poorness of such milk, which is often set up as a defence in prosecutions for watered milk.

The subjoined table gives a selection of the results tabulated by the authors :

Origin of Milk.	Age of Cow.	Age of Milk.	Total Solids in Milk.	Total Solids in Whey.	Soluble Ash	Chlorides as NaCl.	Lactose.	Oleorefractometer Readings corresponding to		
								Lactose.	1 Gramme of Substance other than Lactose.	Total Substances dissolved in Whey.
Vendée	11 years	7½ months	119·8	72·0	3·97	1·42	54·22	33·45	0·38	40·2
,, ...	7 ,,	1 month	129·2	73·20	6·22	1·04	58·50	36·09	0·33	41·0
,, ...	6 ,,	Colostral milk	202·4	67·38	6·30	2·75	37·80	23·32	0·51	38·5
,, ...	4 ,,	9 months	121·0	69·45	5·40	1·37	—	—	—	39·9
,, ...	4 ,,	Colostral	192·2	74·60	5·50	1·83	49·72	30·67	0·47	42·5
,, ...	10 ,,	6 months	152·8	65·85	3·22	1·74	54·22	33·45	0·73	42·0
,, ...	4 ,,	15 days	145·2	70·50	3·22	1·12	55·12	34·00	0·71	45·0
Paris (Dutch cows)	—	—	110·2	61·20	8·25	2·04	45·00	27·76	0·72	39·5
,, ,,	—	—	121·8	69·15	7·87	1·70	51·52	31·78	0·60	42·4
,, ,,	—	—	122·8	69·45	7·27	1·87	50·62	31·23	0·59	42·4
,, ,,	—	—	140·0	63·00	6·22	2·79	47·47	29·28	0·70	40·2
,, ,,	—	—	135·2	65·55	5·32	2·53	48·15	29·70	0·60	40·2
,, ,,	—	—	122·4	65·40	5·92	2·12	51·07	31·50	0·71	41·7
,, ,,	—	—	141·4	66·60	5·47	2·07	51·07	31·51	0·62	40·2
,, ,,	—	—	128·2	67·80	5·47	1·91	55·12	34·00	0·60	41·7
,, ,,	—	—	131·0	66·00	4·27	2·25	50·40	31·09	0·68	41·7

C. A. M.

Analysis of Lemon Syrups. A. Borntraeger. (*Zeit. für Untersuch. der Nahr. und Genussmittel*, 1898, 225.)—The author communicates the methods he elaborated for the analysis of lemon syrup, and the results obtained.

For the detection of sulphuric, hydrochloric, and boric acids a solution of the sample was evaporated with soda, and the residue charred. One portion of the aqueous extract of the residue was then acidified with hydrochloric acid, and a second portion with nitric acid, and the two portions tested respectively with barium chloride and silver nitrate. A third portion was acidified with hydrochloric acid and tested for boric acid with turmeric paper.

Oxalic acid was tested for by heating an aqueous solution with neutral calcium acetate. For the detection of salicylic acid an aqueous solution was shaken with chloroform, the chloroform removed and filtered repeatedly through dry filter-paper until clear, evaporated, the residue taken up with water, and a dilute solution of ferric chloride added.

The heavy metals were tested for by sulphuretted hydrogen and ammonium sulphide. The total acidity was determined by titration, and from the result that due to tartaric acid was deducted, the remainder being reckoned as citric acid.

The tartaric acid was determined by precipitating as acid potassium tartrate and titrating. To a solution of 20 grammes of the syrup was added 5 grammes of potassium chloride, the solution neutralized with solution of potash and made up to 50 c.c. A solution of 5 grammes of citric acid was then added, the solution stirred, allowed to stand overnight, the precipitated acid potassium tartrate washed, first with

a saturated solution of cream of tartar, and afterwards twice with a 10 per cent. solution of potassium chloride, and finally titrated hot.

For the detection of soluble starch, gum, and dextrin, a moderately strong solution was mixed with six times its bulk of absolute alcohol, the alcohol removed, the residue dissolved in water, and alcohol again added, and the same procedure repeated until the residue contained nothing further of a reducing nature. The residue was then dissolved in water, boiled for fifteen minutes with hydrochloric acid, neutralized and tested with Fehling's solution.

For the detection of gelatin an aqueous solution was tested direct with a solution of tannin. As, however, the presence of much citric acid hinders the precipitation, it is advisable to first neutralize with soda. The presence of an excess of soda, on the other hand, redissolves the precipitate.

For the determination and identification of the sugar, a solution was made by dissolving 5 grammes in 100 c.c. of water. For the determination of the reducing-sugar 25 c.c. of the solution were neutralized with soda and diluted to 100 c.c. Ten c.c. of Fehling's solution and 40 c.c. of water were then added and the titration carried out according to Soxhlet's directions. As the solution of the syrup polarized to the right, and contained no saccharose, the results were reckoned as dextrose. The method used for the detection of saccharose depends upon its inversion by hydrochloric acid. Twenty-five c.c. of the 5 per cent. solution were allowed to stand overnight with 2·5 c.c. of hydrochloric acid of 1·1 specific gravity, the solution neutralized with soda, made up to 100 c.c. and titrated. The polarimeter, however, showed that no reducing sugar had been formed, and therefore no saccharose was present in the sample. Though the 5 per cent. syrup solution showed on the day after its preparation a rotation of +21·9 Ventzke (length of tube = 2 decimetres), only 1·5 per cent. of dextrose anhydride, corresponding to a rotation of +4·9 Ventzke, had been found. This led the author to suspect the presence of very impure starch-sugar containing amylin, which was confirmed in the following way: Ten grammes of the sample were dissolved in water, neutralized while hot with precipitated calcium carbonate, filtered hot, the precipitated calcium citrate with the excess of carbonate washed with boiling water, the filtrate cooled and fermented with must. It was then neutralized while hot, evaporated, treated first with lead acetate and then with sodium sulphate, made up to 100 c.c. and filtered. The liquid showed after four hours as well as after twenty-four hours a rotation of +21 Ventzke = +7° 17′ (2 decimetre tube), whilst its power of reducing was equal to 0·65 gramme dextrose anhydride in 100 c.c., corresponding to +2 Ventzke.

The glycerin was tested for by the German official method for the analysis of dry wines, but none was found.

The author has also made analyses of lemon-juice. The following results were obtained:

LEMON SYRUP.

100 Grammes contained	I.	II.	III.
Citric acid	6·75	14·40	5·42
Tartaric acid	10·58	Nil.	10·70
Reducing-sugar reckoned as dextrose ...	—	30·10	38·42
Saccharose	—	Nil.	Nil.
Amylin	—	Present.	Present.
Total extract	—	81·92	80·56
Extract minus citric and tartaric acids and reducing-sugar	—	37·42	26·02
Mineral matter (ash)	—	0·32	0·72
Polarization, Ventzke (length = 2 decimetre tube)	—	+21·9	+19·0

LEMON JUICE.

100 Grammes contained	Ripe Fruit.	Unripe Fruit.
Citric acid, calculated from acidity	7·25	7·70
Citric acid, precipitated as calcium salt	7·28	7·52
Tartaric acid	Nil.	Nil.
Reducing-sugar	0·75	0·21
Saccharose	0·19	0·78
Ash	0·384	0·486
Total extract	8·87	9·30
Extract minus citric acid and varieties of sugar ...	0·68	0·61
Observed rotation	−1·7 (−0° 36′)	+1·3 (+0° 27′)
Polarization corresponding to the saccharose ...	+0·7	+3·0
Polarisation of the reducing-sugar { found	−2·4	−1·7
Polarisation of the reducing-sugar { required ...	−0·9	−0·26

H. H. B. S.

The Composition of Semolina and Food Pastes. Balland. (*Journ. Pharm. Chim.*, 1898, vii., 328-330.)—Semolina is prepared from wheat which contains a large proportion of gluten, as, for instance, that grown in Algiers. It is derived from the less starchy part of the grain, and is distinguished from flour by containing more nitrogenous matter (gluten), but less starch, fat, cellulose, and ash. Under the term "food pastes" are grouped such substances as vermicelli, macaroni, and the small fancy shapes such as crosses, letters, etc., used in soup. These consist of semolina kneaded with boiling water. The following table gives the percentage composition of various semolina products used in France, and contains also for the purpose of comparison the analysis of a rice semolina, of tapioca, and of an artificial tapioca manufactured in France from the potato :

	Water.	Nitrogenous Substances.	Fat.	Starch, etc.	Cellulose.	Ash.
Macaroni (1895) ...	11·60	10·98	0·45	76·05	0·28	0·64
,, (1897) ...	12·00	10·89	0·65	75·70	0·26	0·50
Vermicelli (1896) ...	10·90	11·74	0·50	75·74	0·38	0·74
,, (1897) ...	10·00	12·51	0·80	75·51	0·28	0·90
Pâtes d'Italie (1897) ...	10·40	12·51	0·80	75·23	0·30	0·76
Semolina (1895) ...	9·20	13·50	0·85	75·45	0·50	0·50
,, (1896) ...	9·20	10·42	0·55	78·63	0·45	0·75
,, (1897) ...	10·50	11·96	0·60	75·79	0·50	0·65
Rice semolina (1898) ...	10·80	7·34	0·30	80·96	0·40	0·20
Tapioca, foreign (1897)	12·80	0·00	0·20	86·88	0·08	0·04
,, French potato (1897) ...	16·00	0·45	0·15	82·95	0·00	0·45

C. A. M.

Determination of Tannic Acid in Coffee. H. Trillich and H. Göckel. (*Zeit. für Untersuch. der Nahr. und Genussmittel*, 1898, 101.)—The authors use the following method: 3 grammes of coffee are boiled four times for half an hour with water, the extracts mixed together and made up to 1,000 c.c. After filtering (linen is first used, and afterwards paper), 400 c.c. of the clear filtrate are taken, and 1 c.c. of solution of basic lead acetate added. The whole is allowed to stand overnight, and is then filtered. The precipitate is washed, decomposed by sulphuretted hydrogen, the excess of this gas boiled off, the solution filtered, the filtrate evaporated, and the residue weighed.

A sample of raw New Granada coffee examined by this method and by Bell's and Krug's methods respectively gave the following results:

	I.	II.	Mean.
By the above method ...	11·86 per cent.	10·88 per cent.	11·37 per cent.
By Bell's method	4·36 ,,	6·25 ,,	5·32 ,,
By Krug's method	11·12 ,,	11·50 ,,	11·31 ,,

The authors find that each time the solution is evaporated to dryness a portion of the tannin becomes insoluble, and therefore evaporation and drying should be avoided until the final stage of the process is reached.

In roasted coffee the greater part of the tannic acid appears to exist in a combined state, and the authors have not succeeded in devising a method for its determination in this case. H. H. B. S.

The Detection of Small Quantities of Cotton-seed Oil in Edible Oils. M. Tortelli and R. Ruggeri. (*Moniteur Scientif.*, 1898, xii., 335-338.)—The authors recommend the following modification of Bechi's test, as being much more sensitive and reliable than the ordinary methods. Five grammes of the oil are saponified with 30 c.c. of an alcoholic solution of potash (60 grammes in 1 litre of 90 per cent. alcohol), and the soap solution neutralized with 10 per cent. acetic acid, and poured in a thin stream into a hot solution of lead acetate (50 c.c. of a 10 per cent. solution with 250 c.c. of water). The resulting lead soap is washed with hot water, dried with filter-paper, and extracted for about twenty minutes with ether under a reflux

condenser. The ethereal solution is transferred to a separatory funnel, shaken with 60 c.c. of 10 per cent. hydrochloric acid, and allowed to stand for some time. The lower layer is run off, the shaking with hydrochloric acid repeated, and the ethereal layer well washed with water, and finally filtered. After evaporation of the ether, the residue of fatty acids is mixed in a test-tube with 10 c.c. of pure alcohol (90 per cent.) and 1 c.c. of a 5 per cent. aqueous solution of silver nitrate, and the tube placed in a water-bath at 70° to 80° C. In the case of pure olive-oil or of olive-oil mixed with other edible oils, but free from cotton-seed-oil, the liquid remains clear and unaltered even after fifteen minutes, or even after several hours. But if as little as 1 per cent. of cotton-seed-oil be present, the reduction commences immediately, and is complete after a few minutes.

The paper contains tables giving the results of the authors' experiments with olive, earthnut, sesame, maize, and colza oils alone, and when mixed with variable proportions of cotton-seed-oil, all of which bear out their statement as to the delicacy of the test. C. A. M.

The supposed Decolorization of Red Wine by Means of Potassium Permanganate. L. Hugounenq. (*Journ. Pharm. Chim.*, 1898, vii., 321-323.) — A sample of white wine examined by the author gave the following analytical results:

Alcohol	7·13 per cent.
Extract (in vacuo)	22·27 per litre.
Ash	3·59
Alkalinity of ash, as potassium carbonate ...	1·16
Potassium sulphate	1·14
Acidity (total), as H_2SO_4	4·25
,, volatile, as $C_2H_4O_2$	1·23
Reducing substances (as glucose)	1·47
Glycerin	1·07

The ash left on incineration was a red, spongy residue, which gave strongly all the reactions for manganese, and on analysis it was found to contain 0·59 gramme as MnO calculated on 1 litre of the wine.

The author's explanation of the presence of so much manganese is that the wine was originally a common red wine, decolorized by means of animal charcoal and potassium permanganate.

For the rapid detection of manganese in such products the following process is recommended: Ten c.c. of the suspected wine are shaken with 1 or 2 c.c. of soda-lye and 1 c.c. of hydrogen peroxide solution. An immediate mahogany brown coloration is obtained with the adulterated wine, whereas in the case of normal white wines only a yellow tint appears when they are subjected to this test. C. A. M.

On the Presence of an Alkaloidal Principle in Natural Wines. G. Guerin. (*Journ. Pharm. Chim.*, 1898, vii., 323, 324.)—In 1868, Oser (*Bull. Soc. Chim.*, x., 295) isolated from the products of the fermentation of sugar by beer-yeast an alkaloid with a composition corresponding to the formula $C_{13}H_{20}N_4$. Its hydrochloride dried

in vacuo formed a hygroscopic white mass, turning brown on exposure to the air. It was suggested that the same alkaloid might be found in wine, beer, etc.

The author has examined a large number of different wines, and in each case has succeeded in obtaining evidence of the presence of an alkaloidal principle, in the following manner: One litre of the wine, after the addition of a crystal of tartaric acid, was evaporated on the water-bath until all the alcohol had been expelled. The liquid was cooled, made slightly alkaline with potassium hydroxide, and extracted with ether. The residue left on allowing the ethereal extract to spontaneously evaporate was dissolved in several drops of water made slightly acid with sulphuric acid, and this solution gave all the general reactions for alkaloids. C. A. M.

ORGANIC ANALYSIS.

Determination of the Solidification-Point of Commercial Paraffin. R. Kissling. (*Chem. Zeit.*, xxii., 209.)—After discussing the official method of the German mineral-oil industry, and comparing it with the usual English method, the author proceeds to describe his own plan for determining the solidification-point of paraffin. The apparatus consists of a glass cylinder 12 centimetres high by 8 centimetres diameter—a beaker with the bottom cracked out answers the purpose—on which rests a cover having a hole in it, of a size to allow a beaker 8 centimetres high by 5 centimetres diameter to pass through and be supported by the rim. The determination is made as follows: 50 grammes of the sample are placed in the beaker, and heated in a water-bath to about 10° above the solidification-point. The beaker is then suspended inside the cylinder, its rim resting upon the cover, and the melted paraffin stirred with a thermometer until a thin layer of solidified paraffin begins to form on the bottom of the beaker. As soon as this point is reached, a skin will be seen to form upon the upper surface, whilst the whole fluid, which until now has remained clear, becomes suddenly flocculent. The temperature is then read off. The beaker should be kept covered during the whole course of the determination, from the melting of the paraffin to the final reading of the temperature. The following results show with what exactness the method can be worked:

		Determinations.			
		1.	2.	3.	4.
Sample	I. ...	54·1° C.	54·0° C.	54·0° C.	54·1° C.
,,	II. ...	54·5° C.	54·5° C.	54·4° C.	54·3° C.
,,	III. ...	53·8° C.	53·7° C.	53·8° C.	53·9° C.
,,	IV. ...	52·0° C.	52·0° C.	52·1° C.	52·1° C.
,,	V. ...	53·2° C.	53·3° C.	53·2° C.	53·4° C.

H. H. B. S.

On the Fractional Saponification of Oils and Fats. R. Henriques.—(*Zeit. angew. Chem.*, 1898, 338-345.)—By means of his method of cold saponification (ANALYST, xxi., 67 and 192) the author has thrown light upon the changes which

occur during the saponification of oils and fats. On saponifying saturated glycerides of known composition, such as tribenzoin, and a mixture of stearin and palmitin, with one-third of the alcoholic potash required for complete saponification, it was found that the glycerides were converted into the ethyl esters of the respective acids, whilst the whole of the glycerin present was found in the lye. Even with so little as 10 per cent. of the requisite alkali, nine-tenths of the glycerides were converted. For the separation of the esters from unchanged glycerides, advantage was taken of the fact that the former are volatile in a current of steam at 250° to 270° C., whilst the latter are only very slowly decomposed into fatty acids, which can readily be separated from the distillate. The apparatus recommended consists of an ordinary fractionating flask, whose outlet tube is connected with a Liebig's condenser. The flask is fitted with a cork with two holes, through one of which a thermometer passes, whilst the other is for a tube which conducts dry steam to the bottom of the flask. Between the steam generator and this tube is a small copper tube inclined at a slight angle upwards. This is heated with a small flame, and effectually prevents any particles of water from being mechanically carried into the flask. It is stated that with this apparatus 100 grammes of an ester can be distilled in from twenty to thirty minutes, and that the temperature is readily maintained constant.

For the experiments on unsaturated glycerides the following oils were chosen as types: (1) linseed, (2) olive, almond and rape, (3) castor. In each case ethyl esters were obtained by saponification with as little as 15 per cent. of the theoretical alcoholic potash, whilst practically the whole of the glycerin was liberated. No difference in the iodine and saponification value of the esters was observed on fractional saponification with varying quantities of alkali (33½ to 5 per cent.), and from this the author concludes that fractional saponification is of no value as a means of differentiating the fatty acids present in a given oil.

The ethyl esters from linseed-oil, separated by distillation and dried in a current of carbon dioxide, showed the following constants: Saponification value, 180·8; molecular weight, 309·7; iodine value, 168·4; acid value of liberated fatty acids, 198·7; molecular weight, 281·8. As was also observed in other cases, the difference between the molecular weights of the esters and of the fatty acids closely approximated to 28.

On distilling the esters obtained by saponifying linseed-oil with only 10 per cent. of the necessary potash, the glycerides left in the flask gave the following constants: Acid value, 1·1; saponification value, 185·8; iodine value, 174·4; acetyl value, 51·7. These figures correspond to a mixture of di- and triglycerides, but further investigation is required to determine whether an impure diglyceride was really present, and, if so, whether it was formed during the saponification, or during the distillation with steam.

By using methyl alcohol or amyl alcohol in place of ethyl alcohol, the corresponding methyl and amyl esters are formed. The latter are much less volatile than the ethyl esters, and the temperature in the distilling-flask must be raised to 270—280° C.

The ethyl esters of linseed fatty acids are very unstable, and readily oxidizable on exposure to air, finally yielding a substance insoluble in petroleum spirit. The changes which occur are illustrated by the following figures:

	Acid Value.	Saponification Value.	Iodine Value.	Acetyl Value.
Two days old	0	181·7	170·4	42·5
About three weeks	42	213·6	—	85·1
About six weeks	—	220·4	83·5	104·9
Substance insoluble in petroleum spirit ...	—	271·9	36·1	—

The author suggests that the increase in the saponification value is due to a disruption of the molecule and the formation of lower fatty acids. So long as the oxidized esters are soluble in petroleum spirit, they do not react with phenylhydrazine, but a marked reaction is shown by the insoluble substance. This is attributed to the presence of aldehydic or ketose groups. Unlike the linoxyn formed by linseed-oil on drying, the oxidation products of the esters are not solid products.

The distilled esters obtained from almond-oil gave the following constants: Saponification value, 185·0; molecular weight, 302·7; acid value of liberated fatty acids, 204·6; molecular weight of fatty acids, 273·7; iodine value, 91·1. An experiment in the case of rape-oil proved that the same changes occurred during hot saponification.

The glycerides of castor-oil were still more readily converted into esters than those of the other oils examined. The distilled ethyl esters had a saponification value of 174·9, and an iodine value of 84·1. They were less viscous than the original oil, and completely soluble, though not readily so in petroleum spirit. Their taste and physiological properties were found by Dr. Aronson to be the same as those of castor oil.

From these experiments, the author concludes that during the saponification (cold or hot) of all triglycerides with alcoholic potash, the ethyl esters of the acids present are first formed as intermediate products, with the complete liberation of the glycerin. Further investigation is required to determine to what extent the rule applies to esters other than glycerides. In the case of waxes an analogous behaviour is probable. C. A. M.

Contributions to the Analysis of Fat. W. Fahrion. (*Zeit. angew. Chem.*, 1898, 267-273.)—For the simultaneous determination of the saponification number (by Henriques' method) and of the amount of unsaponifiable matter the author recommends the following process: From 3 to 4 grammes of the oil are dissolved in 25 c.c. of petroleum spirit, 25 c.c. of approximately normal alcoholic potash or soda (containing not more than 5 per cent. of water) added, and after standing twenty-four hours the solution titrated with semi-normal acid, and the saponification number calculated from the results.

The neutral soap solution is washed into a separatory funnel and extracted with successive portions of 20 to 30 c.c. of petroleum spirit. The separate extracts are washed with 10 c.c. of 50 to 60 per cent. alcohol, the washings being returned to the funnel, and the extraction continued until the petroleum spirit leaves no residue on evaporation.

The following examples are given in illustration of this method:

	Saponification No.	Unsaponifiable Matter Per cent.
Beef fat	... 198·3	0·17
Lard	... 196·8	0·12
Palm-oil	... 199·7	1·07
Olive-oil	... 189·0	1·07
Cotton-oil	... 191·1	1·05
Rape-seed-oil	... 173·8	1·48
Castor-oil	... 178·6	0·59
Colophony	... 173·9	3·85

With reference to these results, the author remarks that the unsaponifiable matter of the palm-oil contained the coloring matter present, which, as has before been observed, became bleached on exposure to the air. Concordant results for the unsaponifiable matter, but not for the saponification number, were obtained with liver-oils, whilst there were considerable variations in both particulars in the case of wool-fat. This is accounted for by the fact that the latter contains lactones which on the addition of potash are converted into the salts of the corresponding acids, the conversion being the more complete the longer the contact with the alcoholic lye, and the saponification value being correspondingly raised. On subsequently treating the soap with dilute alcohol, a considerable quantity of these salts is reconverted into lactones, thus causing the variation in the amount of unsaponifiable matter.

The unsaponifiable matter from the various oils was tested according to Liebermann's method in the following manner: From 10 to 20 milligrammes were dissolved in about 3 c.c. of acetic anhydride, and 2 or 3 drops of concentrated sulphuric acid added to the cold solution. With the unsaponifiable substances from palm-oil, olive-oil, cotton-oil, rape-oil, castor-oil, and liver-oil a bright blue colour appeared, which rapidly changed to green, and finally, after an hour or more, to yellowish-brown. In the case of wool-fat the blue colour was not obtained, but the green was very bright.

In order to determine whether this might serve as an indication of the presence of cotton-oil in lard, the unsaponifiable matter was extracted from a mixture containing 10 per cent. of the oil and tested as above. There was an immediate blue colour, changing into green, whilst in the case of pure lard the blue was extremely faint, and only after a considerable time did a faint green tint appear. A similar difference was found in the fatty acids when examined by this test. Those from pure lard gave a faint yellow colour, and only after the lapse of fifteen minutes was a slight green coloration to be detected. On the other hand, the fatty acids from the lard containing the 10 per cent. of cotton-oil gave a yellow colour, which after two minutes changed into a well-marked green.

With the unsaponifiable matter from colophony Liebermann's reaction gave a characteristic violet colour, which rapidly disappeared (*Cf.* preceding abstract).

C. A. M.

The Liquid Fatty Acids of Seal Fat. E. Ljubarsky. (*Jour. prakt. Chem.*, 1898, lvii., 19-27.)—The fatty acids of a sample of Caspian seal fat, when converted into lead salts and treated with ether, yielded 17 per cent. of a solid fatty acid, apparently from its melting-point, palmitic acid, and 83 per cent. of liquid fatty acids, which were found to consist of oleic and physetoleic acids, the latter being the larger constituent, and yielding dihydroxy-palmitic acid on oxidation with alkaline permanganate. In the examination of the oxidation products, it was found that dihydroxy-palmitic and dihydroxy-stearic acids could combine to form a molecular compound, with physical properties quite distinct from those of its separate components. Thus, it melted at 120° to 124° C., solidified at 120° C., and was soluble in hot water, whereas dihydroxy-stearic acid is nearly insoluble. Attempts were made to obtain margaric acid by the action of phosphorus and iodine in the presence of water on this apparently dihydroxy-margaric acid, and subsequent reduction of the fatty iodide with nascent hydrogen, but without success, as the reduction products were invariably found to consist of a mixture of palmitic and stearic acids. C. A. M.

On the Precipitation of Proteids. H. Schjerning. (*Zeit. anal. Chem.*, 1898, xxxvii., 73-87.)—The facts that the same amount of proteid nitrogen is precipitated by ammonium sulphate and by zinc sulphate (Bömer, ANALYST, xxi. 16), and also by uranium acetate and phosphotungstic acid, although under different conditions (*Zeit. anal. Chem.*, xxxiii., 294), led the author to make experiments to determine whether similar salts of analogous metals would precipitate the same amount of nitrogen under similar conditions. Parallel determinations were made on the lines indicated in the author's previous paper (ANALYST, xxiii., 105), using solutions of diastase, peptone, egg albumin, milk and beer, and the following were the mean results obtained throughout the series:

	NITROGEN PER CENT. PRECIPITATED BY									
CHLORIDES.		ACETATES.		SULPHATES.						
Tin. ($SnCl_2$)	Lead.	Iron (Fe_2O_3).	Manganese (Mn_2O_3).	Magnesium.	Zinc.	Cadmium.	Nickel.	Iron (FeO).	Manganese (MnO).	Sodium.
5·1	6·4	63·0	60·8	41·8	40·0	38·2	35·5	37·5	44·3	43·4

The results obtained with chromium acetate were much too low, but this the author explains on the ground that the complete analogy between chromium and iron is doubtful.

Although copper salts present some analogies with salts of the magnesium group, precipitation with copper sulphate gave very much lower results either with or without saturation. For example:

	Magnesium.	Copper.	Iron.
Beer	17·4	4·5	—
Egg albumin	94·8	81·3	82·8

From this it appears that copper sulphate and ferrous sulphate precipitate almost the same quantities of nitrogen. Three metals which form a naturally ascending but

not analogous series are lead, copper, and mercury. With the acetates of these metals the following percentages of nitrogen were precipitated:

	Lead Acetate.	Copper Acetate.		Mercuric Acetate.	
		Cold.	Boiling.	Cold.	Boiling.
Beer	16·0	19·9	25·4	40·1	43·5
Wort	20·8	20·8	24·3	47·0	46·3

With regard to the influence of the acid, it was found that for the same metal the acetate precipitates more nitrogen than the sulphate, and the sulphate more than the chloride. With the latter the largest precipitation took place in the cold solution; with the other salts on boiling.

The acetates of calcium and strontium only precipitated 84·4 and 85·4 per cent. of the nitrogen of egg albumin respectively. Generally speaking, the precipitating power appeared to increase with the atomic weight in the analogous series.

The noble metals are unsuitable for the precipitation, chiefly on account of the readiness with which their salts are reduced to the metallic state.

The following facts have been established with regard to metals suitable for this purpose:

1. The sulphates and chlorides precipitate at most only true albumins, and these often incompletely.

2. The acetates of the magnesium group and of the extended magnesium group precipitate only true albumins. The precipitating power appears to rise with the atomic weight.

3. The acetates of lead and its analogues (?) precipitate all the proteids up to the pro-peptones (albumoses).

4. The acetates of the analogous oxides, Fe_2O_3 and Mn_2O_3, precipitate all proteids up to the real peptones.

5. Uranium acetate and phosphotungstic acid precipitate all the proteids, being examples of the analogous metals uranium and tungsten, although not of the same salts. Uranium acetate can also precipitate some of the ammoniacal nitrogen in the presence of phosphoric acid, whilst phosphotungstic acid precipitates the whole of the nitrogen in that form.

With regard to mercury, which the author looks upon as a transition metal between the heavy non-noble and the noble metals, the following particulars are given: The chloride precipitates all proteids up to the pro-peptones; in other words, the same amount of nitrogen as is precipitated by lead acetate. Mercuric acetate precipitates all the proteids, and, in addition, more or less of the amide nitrogen present. It is well known that mercury forms amido compounds insoluble in acetic acid. Hence the author considers this salt even less suitable than copper acetate for the quantitative precipitation of proteids.

The author draws a distinction between precipitations carried out in the method described and precipitations by saturation. The former he attributes to a definite chemical reaction, whilst in the latter case there is probably a withdrawal of the solvent. He considers it probable that all easily soluble sulphates would precipitate

all albumins and pro-peptones if added to saturation in the presence of a little acetic acid.

From the positive results obtained with the salts of analogous metals, and the negative ones with metals of non-analogous series, the author concludes that the proteids precipitated by the former are definite individuals. C. A. M.

Testing Varieties of Glue. R. Kissling. (*Chem. Zeit.*, xxii., 171.)—The author has examined two recent methods for testing glue, viz., Stelling's (*Chem. Zeit.*, xx., 461) and Fels' (*Chem. Zeit.*, xxi., 56). Stelling's method consists in the determination of the matter other than glue, and depends upon the fact that when glue is treated with alcohol of 72 per cent. it gives up to it a certain quantity of fatty matter and some substances of an extractive nature, the gelatin being insoluble. Fels' method consists in the determination of viscosity. The two methods were compared with that of the author previously published (*Chem. Zeit.*, xvii., 726), which consists in determining the firmness of the jelly by observing the length of time taken for rods of glass, zinc, and brass to sink through it. The results were not very satisfactory, though a certain parallelism was noticeable between the jelly firmness and viscosity, and Stelling's process showed that those glues yielding the firmest jellies contained the smallest proportion of matter not gelatin.

In most cases the value of glue depends so greatly upon the purpose for which it is intended to be used that a knowledge of this is requisite in judging of its suitability. If freedom from smell combined with firmness and freedom from acidity be required, the best leather-glue should be chosen; if, however, adhesive property be the only consideration, the cheaper bone-glues suffice. H. H. B. S.

Analysis of Raw Materials containing Tartaric Acid. J. Schäfer. (*Chem. Zeit.*, xxii., 255 and 269.)—Of all methods that have been proposed for the determination of tartaric acid in lees, argol, tartar, etc., the most widely adopted is the hydrochloric acid method of Goldenberg, of which the following is a brief description: The tartaric acid in the sample is set free by hydrochloric acid, excess of potassium carbonate added, and the solution boiled to convert the tartaric acid into its neutral potassium salt. The solution of this is then concentrated by evaporation to about 10 c.c., precipitated by glacial acetic acid in the presence of alcohol of 96 per cent., and the precipitate filtered off, washed, dissolved in hot water, and titrated hot with soda solution. It appears, however, as the following experiments show, that this method yields results above the truth: Two 50 c.c. portions of the same hydrochloric acid solution being taken, each was treated exactly as Goldenberg directs, except that in one case alcohol of 30 per cent. was used instead of alcohol of 96 per cent. After standing for six hours each was filtered, washed, and titrated. An experiment was then made to ascertain how much acid tartrate would remain in solution in 30 per cent. alcohol. To this end 1·5 grammes of pure cream of tartar were treated as directed by Goldenberg, with the exception that 30 per cent. instead of 96 per cent. alcohol was used. Now, if to the quantity of tartaric acid found on using alcohol of

30 per cent. be added the quantity found in solution in the alcohol, the total should correspond with that found on using alcohol of 96 per cent. With the exception, however, of high-testing or good samples, this was found to be almost invariably not the case. A few examples are given in the following table:

Material examined.	C.c. $\frac{N}{10}$ Soda Solution required when Alcohol of 96 per cent. was used.	C.c. $\frac{N}{10}$ Soda Solution required when Alcohol of 30 per cent. was used.	C.c. $\frac{N}{10}$ Soda Solution corresponding to the dissolved Tartrate.	C.c. $\frac{N}{10}$ Soda Solution corresponding to the total Tartrate.	Difference between the number of c.c. Soda Solution used.	Difference in per cent. of Tartaric Acid.
Sicilian wine lees ...	49·10	41·98	2·05	44·03	5·07	2·53
,, ,, ,, ...	61·80	57·00	1·50	58·50	3·30	1·65
Portuguese wine lees	59·60	56·21	1·50	57·71	1·89	0·94
,, ,, ,,	53·22	50·60	1·60	52·20	1·00	0·50
Portuguese argol ...	50·07	48·07	1·60	49·67	0·40	0·40
,, ,, ...	50·53	49·00	1·50	50·50	—	—
French wine lees ...	45·80	41·94	1·80	43·74	2·06	1·03
,, ,, ,, ...	50·32	47·87	1·50	49·37	0·95	0·47
Spanish wine lees ...	60·96	56·88	2·00	58·88	2·08	1·04

The high results obtained when alcohol of 96 per cent. is used appear to be due to the separation of a fatty body, which combines with soda, and so vitiates the titration. Its separation with the precipitate increases in proportion to the length of time the solution is allowed to stand after the addition of the acetic acid and alcohol. The difficulty, however, can be almost, if not completely, overcome by extracting the fat from the sample with alcohol and ether previous to the determination, which is shown by the following results:

Material examined. Extracted previous to the determination with Alcohol and Ether.	Per cent. of Tartaric Acid found on filtering immediately.	Per cent. of Tartaric Acid found on standing for twelve hours.	Difference per cent.
Italian wine lees	29·33	29·38	0·05
Spanish wine lees ...	25·27	25·37	0·10
,, ,, ,, ...	28·60	28·70	0·10
Algerian wine lees ...	22·32	22·32	

The choice of an indicator for the titration is also an important matter. When azolithmin is used, the change of colour takes place very slowly—much more slowly, in fact, than in the titration of mineral acids, owing to the formation of Rochelle salt, and the larger the quantity formed, the slower the change. This holds good in the titration of chemically pure acid potassium tartrate, but in the case of lees and tartars the indication is further vitiated by the presence of phosphates.

The following general conclusions are drawn:

1. Feebly red litmus-paper should be used as the indicator.

2. After the addition of the acetic acid and alcohol, the solution should be well stirred and immediately filtered off.

3. The risk of too high results on standing can be minimized by extracting the fat by alcohol and ether previous to the determination. H. H. B. S.

Determination of Ash in Crude Sugars and Saccharine Liquids. G. Morpurgo. (*Chem. Zeit.*, xxii., 257.) The following process has been found suitable for the determination of the ash in Samos wine, which contains up to 20 per cent. of sugar. The residue left on evaporation is mixed with about one-third of its volume of hydrogen peroxide, and carefully evaporated over a naked flame until the mass becomes quite brown. After cooling, a few drops more of hydrogen peroxide are added, and the mass dried and ignited. In many cases quite a low red heat is sufficient to convert the residue into a pure white ash. Should this, however, not be the case, a little hydrogen peroxide is dropped upon the parts where the incineration is not complete, and the residue again dried and ignited.

The action of the hydrogen peroxide is mechanical as well as chemical. Small bubbles of oxygen being liberated throughout the mass render it porous and easily consumed at a comparatively low temperature, and of course the enclosed oxygen assists the combustion.

The method is also applicable to the incineration of drugs. H. H. B. S.

Two Colour Reactions for Pyruvic Acid. L. Simon. (*Bull. Soc. Chim.*, 1898, xix., 294, 295.)—Pyruvic acid ($CH_3.CO.COOH$) gives with potassium and sodium nitroprusside a strong violet-red coloration. One c.c. of the solution containing the acid is mixed with the same volume of a solution of potassium hydroxide (specific gravity 1·15 to 1·20), and several drops of a freshly-prepared and fairly-concentrated solution of sodium nitroprusside added. When the acid is only present in traces, the solution should be compared with the alkaline solution of nitroprusside, which itself has a yellow tint. In this way it is possible to detect 1 part of pyruvic acid in 50,000.

The addition of acetic acid to the coloured solution makes the shade a more pronounced violet, but an excess of acetic acid dispels the colour.

The salts and esters of pyruvic acid give the same reaction, but the compound with phenyl-hydrazine gives a negative result. Phenyl-glyoxylic ($C_6H_5.CO.COOH$) acid and its derivatives do not give any coloration, but the reaction is obtained with many other substances (aldehydes, ketones, acetone, etc.). It is, in fact, Legal's reaction, in which, according to Denigés, the coloration is, as a rule, due to the $CH_2.CO$ group, when the carbonyl is not united directly to a certain number of monovalent radicles. Thus, this test, though very sensitive with pyruvic acid, is not characteristic of it alone.

The colour obtained, however, on adding ammonia to pyruvic acid and then nitroprusside does not appear to be given by any substances except that acid and its derivatives and by acetophenone, distinguished by giving a very stable blue colour in Legal's reaction. To 1 c.c. of the solution to be tested are added 1 c.c. of a concentrated solution of ammonia and several drops of the nitroprusside. A

violet-blue colour slowly appears with pyruvic acid, the limit of sensibility being about 1 in 10,000. The appearance of the coloration is accelerated by gentle heat, but the colour disappears on boiling, or on the addition of an excess of acetic acid.

On adding amines of the fatty acid series to nitroprusside and pyruvic acid, a pronounced violet colour rapidly appears; on the addition of acetic acid, this changes to blue and then disappears. This reaction is quite distinct from that given with ammonia. The colour appears more rapidly, but is more evanescent, especially in the presence of acetic acid; whereas in the case of the ammonia, the addition of a small quantity of acetic acid makes the colour more pronounced. C. A. M.

Reducing Action of Filter-Papers. H. Jervis. (*Chem. News*, lxxvii., 133.)—The author has made experiments to ascertain the reducing action of filter-paper upon chromate solutions by digesting various descriptions of paper in hot and cold solutions for half an hour.

Each paper (12·5 cm. diameter) was digested in 25 c.c. of a solution of potassium chromate (1 c.c. = 0·01 gramme Fe) diluted to 200 c.c. In one set of experiments the solutions were made acid with 50 c.c. of normal sulphuric acid, and in another set alkaline with 50 c.c. of normal sodium hydrate. The following table shows the potassium chromate left after digestion in terms of cubic centimetres of the original solution:

12½ cm. Paper.	Ash.	Cubic centimetres K_2CrO_4 Hot.		Cold.	
		Acid.	Alkali.	Acid.	Alkali.
English (Ford Mill, 428)	0·0048	23·7	24·8		
Schleicher and Schüll (No. 589)	0·0003	23·8	24·2	25·0	25·0
Thin German (S. and S.)	0·0028	24·2	24·6		
Ordinary German (S. and S.)	0·0028	23·6	24·6	25·0	25·0
Swedish (Munktell)	0·0005	24·6	24·9		
French White	0·0070	24·3	24·7		
French Gray	0·0139	22·5	24·0	24·4	24·7
White Blotting (Ford Mill, 428)	0·0095	22·9			

H. H. B. S.

INORGANIC ANALYSIS.

Determination of Lead in Water. A. Liebrich. (*Chem. Zeit.*, xxii., 225.)—The crude sulphide precipitated by sulphuretted hydrogen is converted into sulphate by incineration and treatment with a few drops of nitric and sulphuric acids. This is then dissolved in a few c.c. of warm potash solution (1 : 10), the solution filtered, made up to 20 c.c., 2 c.c. of ammonium sulphide added, and the lead determined colorimetrically by comparison with the colour of equal volumes of similar solutions containing known quantities of lead. 0·001 gramme of lead in 10 c.c. is a suitable strength for a stock solution. 1 c.c. of this diluted to 20 c.c. will

give a strong brown colour on addition of 2 c.c. of ammonium sulphide. A gravimetric determination can also be made in the solution used for the colorimetric test by warming it and filtering off the lead sulphide, washing with water containing a little acetic acid, incinerating in a porcelain basin, and converting into sulphate by treatment with nitric and sulphuric acids. By working upon 1 litre or more of water, very exact results can be obtained. H. H. B. S.

The Estimation of Cerium in the Presence of other Rare Earths. G. von Knorre. (*Zeit. angew. Chem.*, 1897, 685-688.)—This is based on the fact that the yellow-colour ceric salts are reduced by hydrogen peroxide in the presence of free acid to colourless cerous salts, as in the equation:

$$2Ce(SO_4)_2 + H_2O_2 = Ce_2(SO_4)_3 + H_2SO_4 + O_2.$$

The cold solution of the ceric salt is mixed with an excess of a dilute solution of hydrogen peroxide, of which the strength is known, and when all colour has disappeared the excess of peroxide is titrated back with permanganate.

If the permanganate solution be standardized on an iron solution, the amount of cerium present may be expressed in terms of iron, 56 parts of the latter being equivalent to 140 parts of cerium. It is advisable to use a dilute solution of permanganates (not containing more than 2 grammes of $KMnO_4$ per litre).

Notwithstanding Rose's statement that potassium permanganate is slowly decolorized by a solution of cerous sulphate, the author finds that the end-reaction can be readily recognised. With a freshly prepared acidified solution of a ceric salt the reduction takes place instantaneously, but if the solution has been exposed to the air for some time, as long as fifteen minutes may be necessary for complete decolorization. The results obtained, however, in both cases are identical. By boiling an old solution after the addition of sulphuric acid, and cooling before adding the hydrogen peroxide, the rate of reduction is accelerated, and the reaction takes place almost as rapidly as in a freshly prepared solution.

Either sulphuric acid or nitric acid may be used, but it is essential that the acidification shall take place before the addition of the hydrogen peroxide, since otherwise by-reactions occur and the results are too high. C. A. M.

A Colorimetric Method of determining Silica in Water. A. Jolles and F. Neurath. (*Zeit. angew. Chem.*, 1898, 315, 316.)—This is based on the fact that alkaline molybdates form in the presence of free nitric acid characteristic yellow compounds with silicic acid similar to those produced with phosphoric and arsenic acids. Of the alkaline silico-molybdates, the potassium compound is the most soluble, and its aqueous solution also possesses the most intense yellow coloration. This gradually increases as the temperature rises, and reaches its maximum at 70° to 80° C.

In the application of this reaction to the determination of silica in water, the authors employ a solution of potassium molybdate, prepared by dissolving 8 grammes of the salt in 50 c.c. of water, and adding 50 c.c. of nitric acid (specific gravity 1·2).

One c.c. of this reagent is added to 20 c.c. of the water under examination, and at the same time to each of several glass cylinders of the same dimensions which contain dissolved in 20 c.c. of distilled water known quantities of silica in the form of soluble glass. The cylinders are placed in a water-bath, the temperature gradually raised to 80° C., and the colour produced in the sample of water compared with that of the standard samples.

It is improbable that phosphoric acid would be present in sufficient quantity in 20 c.c. of water to influence the results, whilst arsenic acid would not be met with in ordinary water. In any case, however, a quantity of the water may be evaporated to dryness several times with nitric acid in order to remove silica, the residue treated with water, the solution filtered and made up to the original volume, and 20 c.c. tested with the molybdate solution as above. Should no yellow coloration appear, phosphoric and arsenic acids must be absent; whilst, on the other hand, if a positive result be obtained, the amount may be determined as silica, and deducted from the result previously found.

The results obtained in this way are compared with those given by the ordinary gravimetric method in the subjoined table:

	Direct Colorimetric Determination.		Gravimetric Method.
	Silica in 20 c.c.	Milligrammes per litre.	Milligrammes of Silica per litre.
Deep-well water ...	0·00006 gramme	3	2·9
Spring water ...	Between 0·0004 and 0·0002, but nearer to 0·0004 = 0·00035	17·5	15·6
,, ,, ...	0·0002	10	9·3
,, ,, ...	Between 0·0006 and 0·0004, but nearer to 0·0004 = 0·00045	22·5	21·2

C. A. M.

The Determination of Oxygen Dissolved in Water. Albert Levy and F. Marboutin. (*Bull. Soc. Chim.*, 1898, xix., 149-151.)—The water to be examined is introduced into a double-stoppered pipette, is made alkaline with potash, and a known volume of ammoniacal ferrous sulphate added. Potassium sulphate is formed, and the ferrous hydrate precipitated is partially transformed into ferric hydrate by the oxygen present in the water. To determine the amount of ferric hydrate produced, an excess of sulphuric acid is added to convert both oxides into sulphates, and the residual ferrous sulphate estimated by means of standard permanganate, and deducted from the quantity originally added. In order to avoid the error introduced by the action of the permanganate on the organic matter contained in certain waters, a blank determination is made on the same water. Experiments are described to prove that the temperature and length of time of the reaction have no influence on the results (*cf.* ANALYST, this vol., 52). C. A. M.

Titration of Thiosulphate with Iodic Acid. C. F. Walker. (*Zeits. anorg. Chem.*, 1898, xvi., 99.)—This article consists of a detailed examination into the method lately suggested by Riegler (ANALYST, xxii., 25) for the estimation of thiosulphate by titration with standard iodic acid. The results show that the process is inaccurate; for not only is the appearance of the blue colour at the end of the reaction wanting in sharpness, but the reaction itself is accompanied by some regeneration of thiosulphate, which causes the figures to be largely dependent upon the volume and concentration of the liquids employed, as well as on the time taken in the operation.

The purest iodic acid of commerce often contains an excess of iodine, probably because it consists partly of anhydride. It may be obtained of theoretical composition by recrystallization and drying over sulphuric acid at atmospheric temperatures; while, as stated by Riegler, its solution maintains its original strength unchanged in the dark for at least four months. F. H. L.

Specific Gravity Tables for Hydrofluoric Acid. (*Chem. Zeit.*, xxii., 225.)—The following tables showing degrees Baumé, specific gravity, and strength of solutions of hydrofluoric acid have been prepared at the experimental station of J. L. C. Eckelt, Berlin:

°Bé.	Sp. gr.	HF%.	°Bé.	Sp. gr.	HF%.	°Bé.	Sp. gr.	HF%.
1	1·0069	2 32	16	1·1239	32·78	31	1·2716	66·61
2	1·0139	4·04	17	1·1326	35·15	32	1·2828	68·76
3	1·0211	5·76	18	1·1415	37·53	33	1·2943	70·91
4	1·0283	7·48	19	1·1506	39·91	34	1·3059	73·06
5	1·0356	9·20	20	1·1598	42·29	35	1·3177	75·21
6	1·0431	10·92	21	1·1691	44·67	36	1·3298	77·36
7	1·0506	12·48	22	1·1786	47·04	37	1·3421	79·51
8	1·0583	14·04	23	1·1883	49·42	38	1·3546	81·66
9	1·0661	15·59	24	1·1981	51·57	39	1·3674	83·81
10	1·0740	17·15	25	1·2080	53·72	40	1·3804	85·96
11	1·0820	18·86	26	1·2182	55·87	41	1·3937	88·10
12	1·0901	21·64	27	1·2285	58·02	42	1·4072	90·24
13	1·0983	24·42	28	1·2390	60·17	43	1·4211	92·39
14	1·1067	27·20	29	1·2497	62·32	44	1·4350	94·54
15	1·1152	29·98	30	1·2605	64·47	45	1·4493	96·69

H. H. B. S.

Estimation of Free Sulphuric Acid in Superphosphates. D. Crispo. (*Rev. Chim. Anal. appl.*, v. [24], 482.)—In view of the errors attaching to the only method of determination hitherto published (extraction of the free acids by alcohol and precipitation by barium chloride from a hydrochloric solution), the author recommends the following as giving exact results and enabling small quantities of sulphuric acid to be estimated in the presence of very considerable amounts of phosphoric acid.

The superphosphate is extracted with strong alcohol, and the dry matter left on the evaporation of the solvent is taken up with absolute alcohol. The insoluble

residue then remaining is dissolved in hydrochloric acid, boiled to convert the meta- and pyro-phosphoric acids into the ortho-acid, and after eliminating this latter, the sulphuric acid is determined by means of barium chloride. C. S.

On the Reduction of Sulphurous Acid to Sulphuretted Hydrogen. E. Donath. (*Zeit. anal. Chem.*, 1897, xxxvi., 663-665.)—It is well known that sulphurous acid, itself a reducing agent, is comparatively easily reduced. Thus a hot solution of stannous chloride reacts with sulphurous acid, probably in accordance with the equation, $3SnCl_2 + SO_2 + 6HCl = 3SnCl_4 + 2H_2O + H_2S$. If too much hydrochloric acid be not present, a precipitate of stannic sulphide or of a mixture of stannous and stannic sulphides is obtained. This reaction has the following analytical applications:

Detection of Arsenic and Antimony in presence of large quantities of Tin.—The solution under examination is mixed with a freshly-prepared strongly acid solution of stannous chloride, the mixture heated to boiling, and a solution of sodium sulphite or of sulphurous acid added drop by drop. The sulphides of arsenic and antimony are precipitated whilst the tin remains in solution.

Detection of Arsenic in Vitriol.—From 10 to 15 c.c. of the commercial acid are diluted with an equal volume of water, and treated as above. When arsenic is present, there is a separation of finely-divided arsenic sulphide after warming for some time, whilst in other cases there is only a strong opalescence due to the separation of sulphur. C. A. M.

A New Gas-Volumetric Method for the Determination of Nitrous Acid and other Substances Oxidizable by Hydrogen Peroxide. E. Riegler. (*Zeit. anal. Chem.*, 1897, xxxvi., 665-668.)—This is a differential method which consists in oxidizing the acid with hydrogen peroxide, in accordance with the equation $HNO_2 + H_2O_2 = HNO_3 + H_2O$, and then determining the volume of oxygen yielded by the excess of the reagent in contact with permanganate. A blank determination is made with a similar quantity of hydrogen peroxide under the same conditions, and the difference between the two results gives the amount of oxygen taken up by the nitrous acid. One c.c. of oxygen at 0° C. and 760 mm. corresponds with 0·0034 gramme of N_2O_3. For measuring the gas the author prefers a modification of the Knop-Wagner azotometer.

In making a determination, 5 c.c. of hydrogen peroxide solution containing not more than 1 to 1½ per cent. of hydrogen peroxide are placed in the flask, together with 30 c.c. of water and about 10 c.c. of concentrated sulphuric acid, whilst a few crystals of permanganate (about 0·1 gramme) are placed in the small inner vessel. The flask is then connected with the measuring-tube, shaken until a permanent pink colour is produced, and the liberated oxygen collected and reduced to standard temperature and pressure. Let the volume equal V_0.

The flask is then washed out, and about 30 c.c. of the solution of nitrous acid (which must not contain more than 0·15 per cent. of N_2O_3) introduced, together with 5 c.c. of hydrogen peroxide solution and 1 or 2 drops of sulphuric acid. The flask is shaken, allowed to stand for about five minutes, and then 10 c.c. of sulphuric acid

gradually added. The crystals of permanganate contained in the inner vessel are then allowed to come into contact with the solution, and the gas collected and measured as before. If this volume be taken as v_0, the amount of nitrous acid is obtained from the formula $\frac{V_0 - v_0}{2} \times 0{\cdot}0034$, or $(V_0 - v_0) \times 0{\cdot}0017$.

In a similar manner the amount of iron contained in a ferrous salt can be determined; in which case $(V_0 - v_0) \times {\cdot}00025$ equals the amount of oxygen corresponding to the iron oxidized to a higher state. The results obtained are said to be excellent.

Potassium permanganate and sulphuric acid may be replaced by silver oxide, the oxidation being then carried out as follows: Ten c.c. of the 1 per cent. solution of hydrogen peroxide, 40 c.c. of water, and 15 drops of a 15 per cent. solution of potassium hydroxide are introduced into the flask, whilst in the inner vessel are placed 5 c.c. of a 3 per cent. solution of silver nitrate. The gas is collected and measured and a second determination made, using 10 c.c. of the hydrogen peroxide solution, 40 to 50 c.c. of the dilute nitrite solution, and 1 or 2 drops of sulphuric acid. The amount of nitrous acid is then calculated from the formula $(V_0 - v_0) \times 0{\cdot}0034 = N_2O_3$.

C. A. M.

Sodium Peroxide in the Quantitative Analysis of Coal, Coke, and Asphalt. C. Glaser. (*Jour. Amer. Chem. Soc.*, 1898, xx., 130-133.)—Clark (*Jour. Chem. Soc.*, 1893, 1079) states that the action of sodium peroxide on coal and coke is too violent to allow of its use in their analysis, but the author finds that it can be successfully used for the determination of sulphur in coal, coke, and asphalt in the following manner:

The material is placed in a large silver or nickel dish, and covered with about four times its weight of sodium carbonate, on which is laid a piece of sodium hydroxide of about half the weight of the carbonate. The dish is moved slowly over a small flame, until the evolution of gas has ceased and a half-fused mass obtained. On this sodium peroxide is dusted in successive small quantities until the reaction is over and the carbon has been completely burned away. If necessary, the mass is then completely fused; but this is usually brought about by the heat of combustion, which takes place quietly and without loss by explosion or spurting. The determination of the sulphur is then completed in the usual way. The time required for combustion is said to be about five minutes for coke, ten for coal, and a little longer for asphalt.

C. A. M.

The Analysis of Coal. F. Haber and S. Grinberg. (*Zeit. anal. Chem.*, 1897, xxxvi., 557-567.)—One of the principal difficulties in the determination of the calorific power of coal by estimating the carbon and hydrogen is that the temperature required to burn the methane with copper oxide is often sufficient to melt the combustion-tube. The authors obviate this by making the combustion in the presence of platinized asbestos. The first 3 cm. of the combustion-tube (43 cm. in length) are left blank, then comes a plug of copper (1 cm.), a layer of granular lead chromate (6 cm.), and a second plug of wire gauze (0·5 cm.). Next comes 1 gramme of finely-divided platinized asbestos (10 to 20 per cent.), which is loosely packed so

as to occupy about 10 cm. of the tube, and finally a third copper gauze plug. The remainder of the tube is left empty, and is closed at the end with a rubber stopper fitted with a T-piece, through the horizontal part of which passes a copper wire with a platinum end, bent into a hook.

By means of this the platinum boat can be moved backwards or forwards within the tube. The other end of the combustion-tube is connected with the usual absorption apparatus, and the part of the tube containing the materials and that occupied by the boat is wrapped in thick copper gauze. For the combustion two Tecla burners are used.

About 0·25 gramme of the finely-powdered coal is placed in the boat, which is introduced into the tube whilst a slow current of oxygen is admitted through the T-piece. After several minutes the boat is pushed forward a little, and so on by degrees until it touches the wire-gauze plug. Care must be taken that the coal does not do more than burn with a red glow, otherwise the combustion is too rapid. In the authors' experience the amount of nitric and nitrous acids found in the absorption-tubes was in every case very trifling, the former being invariably less than 0·05 per cent. Sulphuric acid was never found. The table giving the analysis of several different kinds of coal shows that this method gives results in close agreement with those obtained by the ordinary method of combustion with copper oxide.

C. A. M.

MISCELLANEOUS.

THE LATE MR. F. P. PERKINS.

It is with regret that we have to record the death, at the age of sixty-six years, of Mr. Frank P. Perkins, which took place on June 19. The deceased was one of the oldest Public Analysts in the kingdom, having been twenty-six years Public Analyst for the city of Exeter. He contributed a number of papers to the earlier volumes of the Analyst, on the analysis of butter-fat, the determination of organic carbon in water residues, the estimation of nitrates in river water, the discrimination between oxygen absorbed by nitrites and by organic matter, and on other subjects. He was a member of the Society of Public Analysts for nearly twenty years, having been elected in 1880.

THE ANALYST.

AUGUST, 1898.

PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS.

SUMMER MEETING.

THE summer meeting of the Society this year took the form of a visit to the Duke of Bedford's estate at Woburn, for the purpose of inspecting the Agricultural Experimental Station which has been conducted there for many years by Dr. Voelcker, under the auspices of the Royal Agricultural Society of England. On the morning of Tuesday, July 12, a party of about forty (including a large proportion of ladies) met at Euston and travelled to Bletchley, where, after changing trains, they were conveyed to Woburn Sands Station. Conveyances were in readiness, and after a drive of some three miles through very pretty country the main experimental field, called "Stackyard Field," was reached. Here the various plots of wheat and barley were inspected. Some of these had not received applications of any kind for twenty-two years, others had been treated every year with sulphate of ammonia or with nitrate of soda alone, others with mixtures of various chemical fertilizers, others, again, with farmyard manure. The members accordingly were able to estimate for themselves the influence exercised by the different methods of treatment. All the details were explained by Dr. Voelcker as the party moved on past the various plots, and attention was drawn to the most striking features. Each member of the party was, in addition, provided with a pamphlet, giving plans of the various experiments, the treatment to which each plot had been subjected, and the yield over a series of years. After viewing the fields, the company were conveyed to the experimental pot-culture station, where they inspected the laboratory and also a number of experiments, conducted in pots, designed with a view to discovering the effects on agricultural crops of compounds of some of the rarer elements, such as iodine, bromine, fluorine, lithium, manganese, titanium, etc. This department had only quite recently been established, but several results of an interesting and unexpected nature have already been obtained. After this the party sat down to an excellent luncheon, laid out in the large greenhouse belonging to the station, during which, on the proposition of the President (Dr. Bernard Dyer), a vote of thanks to Dr. Voelcker for his kindness was carried by acclamation, Dr. Voelcker saying, in reply, that the inception of the visit was really due to the fact that the Society had this year an "agricultural president." After luncheon the party were conveyed through the beautiful grounds of Woburn Park to Woburn Abbey, which is the principal seat of the Duke of Bedford. Here the

picture and sculpture galleries and other interesting collections were visited. The weather was most propitious, and the general arrangements were well and carefully organized, the result being a most agreeable, entertaining, and instructive day.

SEWAGE ANALYSIS AND STANDARDS OF PURITY FOR EFFLUENTS.

By C. G. Moor.

(*Read at the Meeting, June* 1, 1898.)

Crude sewage as brought to purification works necessarily varies from town to town. It varies in composition according to the presence or absence of abnormal institutions, such as factories, and according to the manner of its collection; for example, as to whether this is from a high level by continuous gravitation, or from a low level by continuous pumping. The latter is a matter of considerable importance, because in the older precipitation processes it was very difficult to deal with sewage that was stale, as it always is when collected from a low-level system, which is pumped during a few hours only of the working day.

Sewage varies in quantity and in concentration, primarily, according to the extent of the water supply in gallons per head of population, and, secondly, according to the rainfall, etc.

In a given town it varies in all respects from hour to hour, and, as a rule, to a less extent from day to day and from season to season. The extent of none of these variations can be stated in advance. In the estimation of the relative efficiency of the purifying processes used at different towns, it is therefore the ratios of the measurable factors of impurity in the effluent to those in the crude sewage from which it was derived, that must be compared so far as it is possible to obtain them.

In the case of sewage farms these are difficult to obtain. The sewage which is spread over a field will not all find its way into the effluent. Some will be absorbed in the ground, and there disintegrated by the action of micro-organisms and of higher plants.

This is the purification process proper, and its extent will vary up to a limit with the extent of saturation of the soil with previous additions of sewage.

Another part may, however, be temporarily arrested, and the interval which elapses before it is washed through may vary, not only with the nature of the ground and of the crops, but with meteorological and other conditions. This is not necessarily a process of purification, and the comparison of the effluent with the crude sewage may be improperly favourable or unfavourable to the process, according to the time at which the effluent was taken. Evaporation from the surface of the sewage-field will in all cases tend to exhibit the degree of purification as less than it actually is, and less to an extent varying with the presence and nature of the drainage and with the temporary condition of the soil in respect to saturation with sewage. Dilution by rain will affect both sewage and effluent, though possibly not to the same extent; but a more serious variable is the height of the ground-water, which may dilute the

effluent to an extent which would altogether vitiate any figures that did not take account of it.

These are only some of the factors affecting sewage-farms, which are liable to vary beyond control and to some extent beyond measurement. They are indeed inevitable in any system in which provision is not made for the uniform treatment of all parts of the crude sewage.

In the artificial processes, whether chemical, electrical, bacterial, or bacterio-mechanical, the difficulties in the way of obtaining comparable figures are considerably less.

In all of them the whole of the sewage is subjected to a uniform treatment, the interval between the inlet and outlet is known, and the factors of variation between town and town, hour and hour, day and day, may be expected to affect the constitution of the effluent no less than that of the sewage, or at least to affect it to an extent so small as not to seriously invalidate the comparison it is desired to make.

The following scheme of observations would be applicable in most cases where the question to be settled is the efficiency of a particular process at a particular place:

Town Variables.

1. Note nature and extent of factories or other abnormal sources of pollution.
2. Chart the water-supply for previous week and for period of experiments, and ascertain that it is normal.
3. Chart rainfall during the experiments and for the previous week.
4. Note the temperature of the air and sewage during experiments.

Works Variables.

1. Ascertain dimensions of works, the length and fall of sewers, and the general structural details of works and sewerage, and whether pumping is required—*i.e.*, are there low-level sewers?
2. Chart the volume of sewage at intervals during the experiments.
3. Where screens are in use, estimate the solids they retain.

Sewage Variables.

1. Take samples at intervals during the first twenty-four hours of sewage and effluent, determine the chlorine and loss on ignition for each, and compare the result. with that of an average sample taken in quantities proportional to the flow for the time being during the working day, measured so as to include the time of maximum impurity. If these factors give sensibly the same results as average samples, the remainder of the investigation can be conducted on average samples. All samples, average or otherwise, must be kept in ice from the time at which they were taken up to the time of examination, and the interval should be as short as possible.
2. The following estimations and observations should be made:

PHYSICAL EXAMINATION.

Smell.

Colour.—Whether any in one inch in white dish.

Turbidity and determination of suspended matter, if present.

CHEMICAL EXAMINATION.

Total Solids.

Mineral Solids.

Loss on Ignition.—I attach considerable importance to this, and shall refer to it later on.

Chlorine.—This figure is of high value in enabling us to identify the effluent with a sample of sewage with which it is desired to compare it. If one goes to a sewage works at 11 or 12 o'clock, when the sewage is foulest, and takes samples of the raw sewage and of the effluent, they will almost invariably not correspond. An example of this is shown in the case of some analyses made for the Manchester Corporation, the figures of which appeared in the *Sanitary Record* for May 27. A sample of raw sewage contained 14·9 grains of chlorine per gallon, whereas the effluent contained only 6·3 grains per gallon, showing that the sewage corresponded to a period of considerable impurity, while the effluent was probably derived from sewage that was not nearly so foul.

Hardness.—Considerable hardness is produced on adding the large quantities of lime sometimes employed, as in Hansen's process, in which lime and sulphurous powder are used, or in other processes where lime and alumina salts are used.

Nitrites.—I regard the presence of these as showing a want of free access of oxygen. They are not often found in effluents unless the latter have been bottled up and kept in the laboratory for some days, in which case nitrites are always present in large quantities.

Nitrates.—If we determine that nitrates ought to be present in definite quantities, it will probably compel those works at which precipitation is practised to add filter-beds to their plant. The presence of nitrates in the effluent is highly desirable, as showing that oxidation has proceeded to some extent, and that true purification has therefore begun, and some figure should be provisionally fixed so that at least a certain quantity of oxidized nitrogen should be present.

Saline Ammonia.—This cannot be objected to as a natural step in the breaking down of nitrogenous matter.

Albuminoid Ammonia.—The lower the quantity of this, the better.

Oxygen Absorbed.—Four hours at 80° F., Tidy's modification of Forschammer's process.

3. Chart the temperature of sewage and effluent. In some bacteriological processes there is reason for supposing that the temperature rises during the operation.

The following supplementary estimations from a bacteriological point of view would add greatly to the interest of the determinations above mentioned:

1. The number of bacteria, in both sewage and effluent, that grow on gelatin, and the number that liquefy that medium. These determinations will be of comparatively little value unless the following estimations are also undertaken, namely, an estimation of those organisms that live at blood-heat, and of the anaërobic bacteria present, which must, of course, be cultured in a neutral gas under special conditions.

In addition, the investigation of the bacterial contents of the effluent in particular would not be complete without a special estimation of the nitrifying bacteria, which

only grow on special media, such as silica jelly. From these considerations it will be conceded that a bacteriological investigation (even if we had the power of duly interpreting the results when obtained) would be no light matter; and in the present state of our knowledge it could not yield results of anything like the practical value that we can derive from a chemical examination.

In regard to the relative importance of the chemical processes, I will not go over ground which is obviously familiar to all of us; but I would draw special attention to the determination of the "loss on ignition" as being one of the most important factors for characterizing a sewage in respect of its richness in organic matters. Personally, I think that for this purpose it is to be preferred as a characteristic estimation to the results of the popular "oxygen-absorbed" process. For instance, cellulose in various forms occurs in considerable quantities in sewage, and this, while it would probably not be attacked by the permanganate used in the "oxygen-absorbed" process at blood-heat, would certainly be liable to undergo bacterial decomposition and to produce offence. No doubt this would come out in the determinations of organic carbon and nitrogen which have been recommended by a high authority for the examination of sewage.

It must be remembered, however, that life is short, and these processes are long; and it is in the belief that their adoption would tend to the limitation of the number of determinations, and, through this, cause a far greater loss in useful information than would be gained by their practice, that I have intentionally omitted them from my list.

By proceeding as I have described, the principal factors affecting the efficiency of sewage purification processes can be obtained, and while the selection of observations which I have made is subject to criticism, there will be probably little fundamental differences between us as to the necessity and sufficiency of such observations or their equivalents. The interpretation of these results is, however, a matter of more complexity, and more likely to excite differences of opinion. On that ground, and rather with the object of drawing discussion than of formulating any final statement, I submit certain considerations which appear to me to be fundamental. In the first place I am convinced that the purification of sewage, so far as it is contemplated by any existing scheme, can only be regarded as a chemical process, and that it is in terms of chemical determinations alone that the results of any process for effecting it can be expressed. I do not lose sight of the fact that the processes (which through the industrious genius of one of our members, and of others who have worked on similar lines, have now been shown to be the most promising) are bacterial; but it must be clearly remembered that the bacteria so involved are merely means for applying biological energy in place of the chemical energy previously employed, and are used for the precisely identical purpose of altering the sewage in its chemical composition, from a substance highly favourable to the growth of putrefactive and some other organisms, into a substance not specially favourable, and perhaps ultimately inimical to their growth. The only terms, therefore, in which a *standard of purification* for a process or a *standard of permissible impurity* in an effluent can be expressed *are necessarily and solely chemical.* The function of bacteriological examination is in no way depreciated by this circumstance. If the

bacteria of sewage were constant in the sewage of all towns and at all epochs, and if the circumstances affecting their survival and metabolic activity were the same in all sewage or, if variant, they equally affected all the organisms essential to bacterial purification, the place for bacteriology in the matter would be confined to the determination of the extent to which effluents favoured or impeded the growth of undesirable organisms, in particular those which directly cause disease, and those which produce offensive organic substances.

This function it must have in any event, and in the present state of knowledge no general statement can be made in regard to the substantial constancy—that is, as to the constancy in particulars material to the purification processes—of the bacterial conditions to which I have referred. It remains, therefore, for bacteriology to determine the relation between the various organisms and the various chemical factors and physical conditions which occur in practice. The problem is one of extreme complexity, and its investigation is the more tantalizing by reason of the probability that many of the organisms normally found may be individually indifferent in the purification process. It is on this ground that I think the present object of such investigation should be rather the examination of these organisms in groups collected under such characters as those which I have selected than the individual identification of particular organisms. Such inquiry in any case has far more than academic interest. It may well turn out that in some cases artificial addition of bacteria may form a part of that process which in the particular circumstances is the most efficient. There remain, again, questions which experience at present has not gone far enough to settle; such, for instance, as the extent, if any, to which the first products of the bacterial energy are in stable or unstable equilibrium, and their liability, or freedom from liability, to become the subjects of secondary disintegrations, and to result in secondary degradation of the effluent. But whatever result such investigations may have, and essential as I regard them to placing the problem of sewage purification on a satisfactory basis, *it is in quantitative terms of the chemical products* which varying bacteria and varying conditions produce that the results of bacteriological investigation must be expressed before they pass over from the domain of experimental science into that of practical application.

In the absence of such data, it is impossible to define any standard either of percentage purification or of maximum permissible impurity which can be regarded as final or constant for all places. Several standards of permissible composition have been proposed; and perhaps it may be convenient if I re-state some of them. There is, first, that of the Rivers Pollution Commissioners:

	In Parts per 100,000.			
	Organic Carbon.	Organic Nitrogen.	Albuminoid Ammonia.	Oxygen Absorbed.
Rivers Pollution Commissioners ...	2·0	0·3	—	—
Derbyshire County Council	—	—	0·1	1·0

In addition, the Rivers Pollution Commissioners' standard requires that a certain degree of alkalinity or acidity is not to be exceeded, that the matter in suspension is not to exceed 3 parts per 100,000 of dry mineral matter, nor 1 part of dry organic matter, that there shall be no visible colour in a stratum 1 inch deep when viewed

in a white dish, nor any metals except calcium, magnesium, potassium, or sodium present to a greater extent than 2 parts per 100,000, and a limit is also set for free chlorine, for arsenic, and for sulphur as sulphuretted hydrogen or free sulphuric acid.

The other standard, suggested by Dr. Barwise, is of a much more practical nature, though it appears to me to be somewhat too severe, and is, in my opinion, rarely attained by any of the processes in common use. The Thames Conservancy, in the upper reaches of the river, recognises, as a "good effluent," any effluent which gives less than 0·2 part per million of albuminoid ammonia. I should be inclined to suggest the following standard as satisfactory for general use:

	Parts per 100,000.
Total suspended matter (not to exceed)	2·0
Albuminoid ammonia	0·2
Oxygen absorbed...	0·75
Total hardness	25·0
Nitrogen as nitrates (not to be less than)	0·75

The amount of nitrates should be insisted on, as showing that true purification has begun, and that there is a store of oxygen that could be drawn on, as a guarantee against possible putrefaction.

I base this standard on the fact that, on the examination of a large number of samples of effluents, I have found no sample which, falling within this standard, became putrefied on keeping either in closed or open vessels. For my own part, therefore, I should at present prefer this standard; but I put it forward with the reservation that any standard must at the present time be regarded as tentative, and be subject to, and likely to undergo, revision in, I hope, no distant future, when more exact data correlating the bacterial, physical, and chemical circumstances are available. In the meantime, however, I think that in this, as in so many other matters in which our profession is concerned, it would add to the authority of all of us, and conduce to the advantage of the public, if we could arrive at some agreement as to the standard which should, for the time being, be adopted in common.

Discussion.

Dr. Dupré said that he totally objected to any universally applicable standard being laid down in the case of sewage effluents. What was or was not permissible depended entirely upon where each effluent was to go to—whether, for instance, it was to be discharged into a comparatively large stream of water, or into a dry, or nearly dry, ditch. The Thames Conservancy had, he believed, several different standards. They would allow to be discharged below the intake of the waterworks an effluent which they would not allow to go into the river above the intakes. A very fair measure of the impurity present in a water or effluent might be obtained by determining the quantity of dissolved oxygen absorbed from the effluent. Pure water remained aerated when bottled up, but water containing sewage de-aerated itself. An effluent which did not appreciably de-aerate itself might be admitted anywhere without fear of putrefaction taking place. As a chemist, he always preferred to rely upon chemical tests, and to do without bacteriology if possible; and this measurement of the oxygen absorbed by the water itself was a fairly good substitute

for a bacteriological examination. Unfortunately, although the actual work could be carried out in a few minutes, the sample had to be allowed to stand for from five to ten days. He thought it of far more importance to agree to some methods of analysis than to lay down standards; he would particularly refer to the determination of the oxygen absorbed by permanganate. This was one of the most useful factors in a water analysis; but, unfortunately, chemists differed very greatly in their methods of arriving at it. Some followed the late Dr. Tidy, and used open flasks kept at the temperature of the laboratory, and hoped that an equal amount of dirt might fall into the flask containing the standard used for comparison, and into the experimental flask; others, like Dr. Rideal, placed their bottles on the top of the water-oven, others actually boiled their acidified solutions. It was, however, very desirable that all should agree to make the determination upon a definite plan. He believed that he had himself been the first to suggest the carrying out of the process in a closed bottle, and at a fixed temperature. In doing so, he had not had in view the question of chlorides being present; but it was found that the use of a closed vessel did away entirely with the difficulties arising from the presence of chlorine when an open flask was used. The determination, in fact, could be made in sea-water as easily as in ordinary drinking-water.

Dr. VOELCKER said that the importance of considering any particular effluent in relation to the water from which it was originally derived extended to other constituents besides the chlorine. For instance, in dealing with effluents from districts where the water was known to be unusually hard, the total hardness would have to be regarded in relation to that of the original water. It was important, also, to know the nature of the suspended matter, and whether it consisted of organic matter or merely of mineral matter. He had not noticed in the paper any reference to a question which he thought was a very important one, and which Dr. Dupré had alluded to, namely, the ratio of the volume of the effluent to that of the stream into which it flowed.

Dr. RIDEAL quoted, as an instance of the unreliability of standards, a case which had been referred to in a paper read by him before the Society of Arts, of a solution of putrid meat extract, diluted with water in the proportion of 1 to 6,000. The liquid was very putrid, smelling strongly of sewage, and was no doubt also a toxic solution containing ptomaines; but it would nevertheless have passed any ordinary standard in regard to free and albuminoid ammonia, oxygen absorbed, etc. What, he thought, was to be aimed at was not so much a fixed standard as a ratio, showing what had become of the nitrogen, and how it had been altered. In the original sewage the nitrogen would be entirely in the organic state. The action of bacteria resulted in the breaking down of this organic nitrogen with the production of free ammonia, followed by the formation of nitrites and nitrates, until finally the nitrogen became entirely oxidized. The total nitrogen in a sewage effluent, therefore, was to be looked upon as existing in four distinct forms, namely, organic nitrogen, free ammonia, nitrous nitrogen, and nitric nitrogen; and it was the ratio of these four different kinds of nitrogen to one another which indicated whether an effluent was satisfactory or not. If the ratio between the first two and the second two was such that the balance of oxygen in the nitrites and nitrates was more than sufficient to oxidize the nitrogen

not yet oxidized, the liquid could not undergo any putrefactive change likely to cause a nuisance. He therefore agreed with Mr. Moor in the importance of ascertaining that the nitric nitrogen was not below a certain amount, but contended that the amount was determined by the amount of the unaltered nitrogen present. He had found the loss on ignition to be a very unsatisfactory figure, yielding no information as to the quantity of organic matter present in sewage after treatment. In some cases it would even increase, instead of diminish, after the sewage had undergone a process supposed to destroy organic matter.

Mr. CHAPMAN said that in his experience it was usually impossible to derive any satisfactory information from the loss on ignition. In very many cases (more especially in impure waters) the loss due to decomposition experienced by the mineral matter, and to the expulsion of combined water, far overbalanced any loss that might be due to the combustion of organic matter. Dr. Rideal had apparently assumed that a reaction would occur between the nitric and nitrous acid, and the ammonia present in a sewage effluent; but he (Mr. Chapman) did not think there was sufficient evidence to show that such a reaction would take place in the dilute solutions in which these substances were found in effluents. It was not always safe to assume that changes which took place in concentrated solutions also took place in very weak solutions.

Mr. CASSAL said it was not possible to lay down rigid standards in matters of this kind. The question was really one that involved the getting of some figures by which the expert might be guided, and he thought that Mr. Moor's object was rather to put before the Society some figures which, upon broad lines, would be of assistance in arriving at conclusions, than to suggest the laying down of any rigid standards. It was quite plain that such matters must be governed by the special circumstances of each particular case. It was dangerous to speak of good, bad, and medium effluents. The object of the Local Government Board in insisting upon the treatment of sewage by passing it through land had obviously been, or ought to have been, the absolute purification of the sewage from objectionable organic matter by filtration through the soil; a purification carried to such an extent that the resulting effluent should be no fouler than the stream into which it was to flow. Theoretically, at any rate, it ought to be assumed that there should not be introduced into any stream or river a liquid which contained in a notable degree a larger quantity of putrescible organic matter than was present in the stream itself. If the stream itself was polluted, the question of special circumstances came in very strongly, and the conclusions of the engineer, as well as those of the chemist, had to be applied in order to determine whether there was any possibility of an increase in the pollution of the stream by the introduction of any given effluent. Apart from this, the aim of the sanitarian should be the introduction into any stream or river of perfectly purified or demonstrably innocuous effluents. He, too, was of opinion that the ignition of water, or sewage "residues," while it was a valuable qualitative test, was not a reliable quantitative one, except when employed in connection with the same set of water, sewages or effluents, as a means of comparison merely among the samples themselves.

The PRESIDENT said that the subject of standards was a most difficult and, indeed, a dangerous one to deal with. The real question in any given case was

probably how far the effluent approximated to the best possible effluent that could be reasonably expected, having regard to local circumstances and to the means which it was reasonably possible to employ. The great absurdity attaching to standards under some conditions had once been shown by a well-known manufacturer at a meeting of the Society of Chemical Industry, at the time when it was suggested that the standards recommended by the Rivers Pollution Commission should be legalised. That gentleman observed that he daily ejected from his works an effluent which in very nearly every particular transgressed the limits laid down, the effluent in question being, however, merely the water of the river on which his factory was situated, which water was pumped into the works for use in cooling the condensers, and afterwards returned in its original condition to the river. The river, however, would be held to be thereby polluted, if the suggested standard were legalised. While, however, it was exceedingly difficult to lay down general standards, it was very desirable that there should, if possible, be some settled means of making the different determinations involved in analyses of this kind. It seemed, for instance, particularly important to decide and agree whether, if an effluent contained suspended matter, it should be shaken up and analysed as a whole, including the suspended matter, or whether it should be filtered, or whether it should be allowed to settle; and also whether, if it was filtered, a close paper or a loose one should be used. Such considerations as these gravely affected the determinations of oxygen absorbed and albuminoid ammonia.

Mr. W. J. DIBDIN desired to compliment the author upon the excellent paper which he had presented to the Society. The question of standards of quality for sewage effluents was perhaps one of the most difficult of all those raised in connection with sanitary problems. It had been suggested that the quality of the effluent should be equal to that of the stream into which it is to be turned. This was at once met by the consideration of the case of an effluent discharged from a sewage farm even in the very best working condition, the outfall from which was on the banks of a stream which had but a few minutes before sprung from, say, the outcrop of the chalk. It would be practically impossible to comply with the prescription, and an exception would have to be made. If one exception, why not two or three? He had long since adopted in his own mind a physiological standard, viz., that the quality of an effluent should be such that fish could live healthily in it. Such an effluent is not a fanciful one, nor is there any difficulty in obtaining it by the employment of suitable means, and these, fortunately, are most economical. Such a definition involves necessarily the absence of poisons and the presence of oxygen. This raises the point as to the quantity of nitrates which should be present, as, according to Mr. Moor, these present a store of oxygen that could be drawn on. This is so; but the statement overlooks the important fact that when water is freely in the presence of air, such as in a stream, etc., it acts like a sponge, greedily absorbing oxygen from the atmosphere as fast as it yields it up to the organisms feeding on the organic matter, so that at some point, determined by the rate at which these organisms require it, a balance is arrived at. If the aeration is kept down very low by the rapidity of the life processes, this absorption of atmospheric oxygen is very rapid, varying in inverse ratio to the degree of aeration. From this consideration it will be evident that, valuable as is the indication afforded by the presence of nitrates, these must not be looked upon solely as a

store of oxygen, and still less as the sole store of that substance for effecting the ultimate purification of the sewage matters. With regard to the nitrites in effluents being only present after these have been kept for some days in bottles, he was afraid that he could not agree with the author of the paper. In the course of a series of special experiments, conducted on the works, Mr. Thudichum found at Exeter that nitrites were present in every sample tested direct as it came from the fine coke-breeze filters. He was in full agreement with the author's suggestion that the lower the quantity of albuminoid ammonia the better, but, unfortunately, this point did not much help the question of standards. With regard to the temperature of the sewage rising in some bacterial processes, this was unquestionably the fact, and was comparable to the familiar instance of the garden hot-bed. In some bacteria beds under his (Mr. Dibdin's) direction the temperature had risen as high as 80° F. With reference to the loss on ignition being an important point, he could not agree with this in all cases. For instance, where a large quantity of mineral salts were sent into the sewage, the loss due to water of crystallization, etc., would often be so great as to swamp all that due to the organic matter, and thus no factor of any value at all from this point of view was obtainable. The value of the relation of the results of analyses must, for the present, at least in the majority of cases, be based upon the personal equation, in which large experience and knowledge of the local conditions as to necessities and possibilities were all involved.

Mr. Moor said that he very much appreciated the sympathetic criticism to which his remarks had been submitted, and, while opinions seemed to vary on points of detail, he could not help thinking that even the variety of opinion which had been expressed emphasized the desirability of having some set of figures on which an effluent could at least be condemned. He had endeavoured very clearly to indicate that the particular set which he had suggested was by no means put forward either as a final standard or as an ideal effluent. Perhaps his meaning would have been made clearer if he had described these figures, as he had in fact intended them, as constituting, not the standard for an effluent which could permanently or in all circumstances be adopted, but the maximum of impurity which should in any circumstances be allowed. By the adoption of this maximum, he in no way intended to suggest that such effluent would in all cases be permissible; for example, he would regard it as quite out of the question for an effluent to be discharged in any quantity above a water company's intake. In making this remark, he desired to add, for fear of misunderstanding, that, in his judgment, no chemical criteria could ever warrant the safety of an effluent above an intake; but that, as the danger would to some extent vary inversely with the chemical purity, he would insist on a much higher standard for effluents supplied to sewage-drinking communities than that which he had discussed in this paper. None of the criticism which had been offered appeared to him to conflict with the adoption of these figures for this purpose, with the single reservation pointed out by Dr. Voelcker—that the hardness of effluents should be fixed in some relation to that of the water-supply. His purpose in including this figure was of course to prevent the undue drugging of the water with chemicals employed in processes of treatment; and he suggested, therefore, as an emendation of the absolute figure which he had at first suggested, the provision that

total hardness of effluent should not exceed that of the water-supply by more than 25 parts per 100,000. He was in entire agreement with Mr. Cassal that at the present time no data existed warranting the description of an effluent as good or indifferent; but he still thought that there would be considerable advantage in having some figures by which in need it could be described as bad, as it followed logically, from what the President had rightly observed, such figures must represent the best effluent which could in all circumstances be attained. He did not understand the President to suggest that the figures which he (the speaker) had proposed were such as were likely to occur in a natural river; and if by artificial pollution a river had been so contaminated as to fall short of his modest requirements, he thought there would be every possible advantage in preventing ingenious and energetic persons, such as the gentleman to whom the President referred, from using such river for any purpose until they had gone up stream and compelled the persons polluting the river to leave off. Taking the determinations which he had suggested individually, he had no hesitation in saying in reply to the President's question that analysis must take impartial account of everything which appeared in the sewage; and, feeling as he did that suspended matter had no right in an effluent to any extent exceeding that which he had suggested, he was convinced that the only discrimination allowed to the analyst was the careful collection of an average sample, which then would be shaken up and analysed as a whole. So far as mineral matter was concerned, his experience was that it settled so much more rapidly than animal and vegetable matter, that the reservation suggested by Dr. Voelcker was not likely to be needed in practice, and he, personally, had met with no case in which it would have modified his proposed criterion. It was to be observed also, from the bacteriological standpoint, that suspended matter was usually far richer in organisms than the liquid conveying it; and as it certainly could be prevented by reasonable measures, he thought it needless to make any allowance for cases in which a substantial part of it might be mineral.

So far as the albuminoid ammonia was concerned, no question appeared to arise as to the figure which he had suggested being too high. He personally preferred the oxygen-absorbed determination to that proposed by Dr. Dupré, because of its greater rapidity; but having regard to the difficulty of attacking all the organic matter in sewage by permanganate, especially where there was much suspended matter, he thought it might be worth considering whether one or other of the higher temperature processes would not be preferable for routine sewage examination to the ordinary Tidy modification of the Forchammer process, which was usually employed. He should have explained that in suggesting loss on ignition as a valuable characteristic of the same order as "oxygen absorbed," he had in mind the successive examinations of the same sewage, such as would occur in the examination of a process; and he quite agreed with Mr. Dibdin that it would not serve as a factor for comparison between sewages of different origins, and for this reason he had not proposed it as part of his suggested criterion. So far as nitrites were concerned, he was bound to say that while he had repeatedly found them in samples of which the examination had been delayed for a short time, he had never found them in freshly-examined samples. He recognised with Mr. Dibdin that there were other sources of oxygen in water than the nitrates,

but seeing that his (the speaker's) figure was only proposed as a minimum, he did not think that this fact would warrant the reduction of the figure of nitrates which should be required. The utility of this factor was evident from Dr. Rideal's meat-extract, which certainly would have been condemned for its deficiency in this respect, and with equal certainty, had it passed through any sewage process capable of bringing it up to the standard in this particular, would have ceased to be offensive. In conclusion, he would reiterate that the whole, if any, of the figures which he had proposed was not to constitute an ideal or permanent criterion, and in that sense perhaps the word "standard" would be as inapplicable to these as to other figures which had been proposed for the same purpose. His figures would unquestionably be superseded by others more severe when data were available for safely defining them. They might even now be supplemented, if it was thought desirable, by Mr. Dibdin's test of the survival of fish, or by Dr. Dupré's requirement of permanent aeration. But the speakers had not suggested, nor was he aware of any sufficient evidence at present available, to justify exclusive reliance on either of these tests; and as, therefore, a chemical criterion was undoubtedly necessary, he appealed to the Society to consider whether it was not for the public interest and for that of the profession that an agreement should be come to at least as to the figures on which all analysts would condemn an effluent.

ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

FOODS AND DRUGS ANALYSIS.

A New Method of sophisticating Coffee. G. Wirtz. (*Zeit. für Untersuch. der Nahr. und Genussmittel*, 1898, 248.)—The author states that coffee is largely sophisticated in Hamburg, Bremen, Antwerp, and Rotterdam, as well as in the places of origin, by the process of washing, coloring, and drying off in centrifugal machines with sawdust, with the result that the furrows of the berries become filled with wood powder, making them a fine white and enhancing the market value of the produce. H. H. B. S.

Paprika (Cayenne) as a Substitute for Alcohol. Kellermann. (*Zeit. für Untersuch. der Nahr. und Genussmittel*, 1898, 247.)—The attention of the author was drawn to a description of spirit which had a sharp taste, and produced a greater sensation of warmth than other competing spirits, though containing less alcohol. The residue left on distilling was found to produce an intense and lasting burning sensation when applied to the mucous membrane of the lips, exactly similar to that produced by "paprika." The addition of an alcoholic extract of "paprika" to common spirits produced a spirit possessed of the same peculiarities. H. H. B. S.

A New Method for the Valuation of Amyl Nitrite and Spirit of Nitrous Ether. **C. E. Smith.** (*Amer. Jour. Pharm.*, 1898, lxx., 273-285).—This is a volumetric process based on the reaction between nitrous and chloric acids:

$$3HNO_2 + HClO_3 = 3HNO_3 + HCl.$$

Spirit of Nitrous Ether.—The following are successively placed in a 100 c.c. flask provided with a loosely-fitting stopper: Ten c.c. of water, 5 c.c. of a cold saturated solution of potassium chlorate, 5 c.c. of the spirit to be tested, and 5 c.c. of 10 per cent. nitric acid. After shaking at intervals for thirty minutes, 10 c.c. of decinormal silver nitrate are added, the flask shaken for a moment, and the excess of silver titrated as rapidly as possible with decinormal potassium thiocyanate, a few drops of a solution of ferrous ammonium sulphate being used as indicator. The end-point of the titration is reached when, after *momentary* shaking, the liquid remains faintly red throughout.

Each c.c. of silver nitrate consumed corresponds to 0·0225 gramme of ethyl nitrite, and if the specific gravity of the sample be taken as 0·84, which is usually approximately correct, the calculation can be shortened by multiplying the number of c.c. by 0·536.

Concentrated nitrous ether may be assayed by diluting with alcohol in definite proportions, and proceeding as above.

The results are invariably higher than those obtained by the usual gaso-volumetric method, but from the results of comparative experiments the author considers the new process the more accurate of the two.

The following results were obtained in the analysis of commercial samples, of which the last was a sample of concentrated nitrous ether, stated to be 90 per cent. ethyl nitrite. It was tested after dilution to 4 per cent.

	Per Cent. Ethyl Nitrite. U.S.P. Method.	Per Cent. Ethyl Nitrite. Chlorate Method.	Per Cent. of Free Acid.	Aldehyde Test.
1.	3·66; 3·70	4·06; 4·01	0·14	Faintly yellow.
2.	4·84; 4·88	5·30; 5·30	0·11	,, ,,
3.	4·28; 4·31	4·58; 4·60	0·45	Dark.
4.	3·95; 4·00	4·38; 4·44	0·15	Straw.
5.	4·35; 4·32	4·81; 4·84	0·31	Dark.
6.	85·50; 85·90	92·60; 92·00	0·46	Straw.

The acid determinations were made with normal soda, without dilution with water. Addition of water or titration with decinormal soda gave considerably higher results, probably through saponification of the esters. All of the samples answered the requirements of the Pharmacopœia (U.S.A.) test for aldehydes, which prescribes that the colour shall not be decidedly brown after twelve hours' contact with alkali.

Amyl Nitrite.—A 100 c.c. flask is partially filled with alcohol and weighed. Five to six grammes of the amyl nitrite are then added, the flask again weighed, and the liquid made up to the mark with alcohol and well shaken. The determination is carried out in the same way as described for nitrous ether, except that double quantities of the reagents and of the alcoholic solution of amyl nitrite are used. One c.c. of decinormal silver nitrate corresponds to 0·0351 gramme of amyl nitrite.

The following table gives the results obtained with commercial samples:

	Per Cent. Amyl Nitrite. U.S.P. Method.	Per Cent. Amyl Nitrite. Chlorate Method.	Per Cent. of Free Acid.	Aldehyde Test.
1.	77·20; 77·0	87·0; 87·4	0·6	Dark straw colour.
2.	76·0; 76·4	86·7; 86·2	0·67	Brownish yellow.
3.	17·3; 18·8	21·0; 18·9	0·76	Deep yellow.
4.	82·7; 83·5	93·9; 93·4	1·36	Dark straw colour.
5.	79·0; 78·4	85·9; 85·3	1·12	Deep yellow.

Of these, No. 3 was from a bottle with an old and defective cork which had been badly attacked by the liquid; Nos. 4 and 5 exceeded the Pharmacopœia limit of free acid, which is 1·08 per cent. as nitrous acid; and none of them were fully up to the standard required by the aldehyde test, which prescribes that on shaking equal volumes of amyl nitrite and normal potassium hydroxide the aqueous layer shall not be more than pale yellow.

The author advises that a blank determination should be made in the assay and an allowance made for the impurities, since potassium chlorate and nitric acid often contain traces of chloride, and nitric acid lower oxides of nitrogen, both of which would affect the results. C. A. M.

Detection of Salicylic Acid in Comestibles. F. A. Genth. (*J. Franklin Inst.*, 1898, cxlv., 228; through *Chem. Zeit. Rep.*, 1898, 97.)—The author considers Krause's distillation process the best for the detection of salicylic acid in articles of food. The substance is rubbed down to a cream with phosphoric acid, water being added if necessary. After a time, the mass is squeezed through a cloth, and 50 or 75 c.c. of the liquid are submitted to distillation, every 5 c.c. of the distillate being tested with ferric chloride. The salicylic acid passes over with the steam, most of it being recovered towards the end of the operation. The methyl-ester reaction is not suited for detecting small quantities of the antiseptic; but by this process Genth has discovered the presence of salicylic acid in various preserves which nominally did not contain it. F. H. L.

The Examination of Methyl Salicylate. Adrian. (*Journ. Pharm. Chim.*, 1898, vii., 122-124.)—Physiological experiments having demonstrated the superiority of methyl salicylate over the natural oil of winter-green, it is important to be able to distinguish between the two, although on account of its higher price the latter is hardly likely to be fraudulently substituted for the artificial product.

The following table gives the comparative results of the fractional distillation of samples of undoubted purity:

Methyl Salicylate.		Density at 15° C.	Oil of Winter-Green.		Density at 15° C.
A little water (5 grammes) distilled over below 220°.			A little water (5 grammes) distilled over below 220°.		
	Grammes.			Grammes.	
Distilled below 220° (nearly all at 219°) ...	110	1·1785			
Between 220° and 222°	105	1·178	Between 220° and 222°	615	1·185
,, 222° and 223°	155	1·179	,, 222° and 223°	190	
,, 223° and 225°	350	1·180	,, 223° and 225°	85	
,, 225° and 230°	185		,, 225° and 230°	50	
Residue	90		Residue	55	
Total ...	1000		Total ...	1000	

The density and boiling-point are valuable tests, and will detect the presence of free methyl alcohol or of ethyl alcohol fraudulently added. The addition of a fixed oil would lower the specific gravity, and a residue would be left on distillation from which, on heating more strongly, acrolein vapours would be liberated.

Methyl salicylate can be distinguished from oil of winter-green by its behaviour with sulphuric acid. On mixing 5 c.c. of methyl salicylate or of one of the distilled fractions (previously well dried) with an equal volume of sulphuric acid, there is no rise of temperature, but the ester assumes a faint yellow colour, with the exception of the fraction distilling below 220°, which does not give the coloration.

On treating the natural product in the same manner, and especially the fraction distilling between 220° and 222°, which contains the terpene, gaultherylene, there is an immediate rose coloration, changing to red and finally to dark brown, and there is also a considerable evolution of heat. C. A. M.

Oil of Sassafras. C. H. La Wall and R. C. Pursel. (*Amer. Jour. Pharm.*, 1898, lxx., 340-342.)—This oil, which is largely employed by soap-manufacturers, is very liable to adulteration. As the boiling-point shows considerable variation in pure specimens, the chief points relied upon in judging of the quality of a sample are its specific gravity, general appearance, and odour. Safrol was formerly used as an adulterant, but its price is now higher than that of the oil. It constitutes normally about nine-tenths of the weight of the oil, and has a specific gravity of 1·108, whilst the specific gravity of sassafras oil varies from 1·07 to 1·09. Oil which has a lower gravity than the lower limit is regarded with suspicion, as it might indicate adulteration with the fractionated camphor oil which is used for that purpose. In cold weather crystals of safrol are deposited, and it may often happen that the upper, middle, and lower portions of the oil are of different gravities, and in such cases the only way to insure a homogeneous mixture is to pour out part of the oil before stirring, and then to agitate very vigorously. The authors refer to an instance in which, after apparently well mixing the whole bulk and leaving no crystals at the bottom, the gravity was found to be 1·055, but after more thorough admixture was 1·07.

The following table gives the maximum, minimum, and mean specific gravity of all the samples examined by the authors from 1896 to June 1, 1898. They represent an aggregate of 10,000 lb. as received from the distiller.

	Maximum.	Minimum.	Mean.
1896	1·0840	1·0500	1·0654
1897	1·0850	1·0610	1·0736
1898	1·0830	1·0450	1·0713

The colour of the pure oil varies from deep yellow to nearly colorless. The artificial oil closely resembles the natural product, and an admixture is not readily detected. C. A. M.

Croton Oil. Javillier. (*Journ. Pharm. Chim.*, 1898, vii., 524-527.)—It is stated that the variations in the constants of this oil, as determined by different observers, are largely due to the method by which the oil was obtained. The author prepared three samples, the first by simple expression, the second by lixiviation with ether, and the third by digestion at 75° C. with 95 per cent. alcohol, the first two methods being those prescribed by the French Codex of 1884. In each case a different yield and a different-coloured product was obtained, *viz.*, (1) 12·5 per cent. of a pale oil; (2) 38 per cent. of a light-brown oil; and (3) 12 per cent. of a very dark-brown oil.

The difference in their physical and chemical properties is shown in the following table:

Solubility in Alcohol.	Expressed Oil.	Oil extracted with Ether.	Oil extracted with Alcohol.
(1 vol. of oil + 2 vol. absolute alcohol.)	Soluble at 75° C.	Soluble at 75°.	Soluble in the cold.
Solidification temperature ...	− 7° C.	− 7° C.	− 8° C.
Iodine value (Hübl)	109	108	91·2
Saponification value	192·9	194·5	260·6
Acid value	27·3	30·9	60·1

The acid value was determined by dissolving the oil in ether and titrating directly with decinormal alcoholic potash. It was found that Henrique's method of cold saponification was not easily effected with croton oil.

The commercial oil is often prepared by the methods given in the Codex of 1837 and 1866, *viz.*, expression followed by digestion with 80 per cent. alcohol. Its iodine value is generally about 102, and the saponification of a sample examined by the author was 205·6. C. A. M.

The Examination of Theobromine. M. François. (*Journ. Pharm. Chim.*, 1898, vii., 521-523.)—The most probable adulterant of theobromine (which has recently been employed in therapeutics) is caffeine, which is considerably cheaper. The author describes the following tests of the purity of the drug:

1. On adding 10 c.c. of a 10 per cent. solution of silver nitrate to a solution made by dissolving 0·1 gramme of theobromine in a hot mixture of 1 c.c. of nitric acid and 2 c.c. of water, a turbidity occurs, which on warming disappears. On cooling, a mass of needle-shaped crystals is deposited.

2. A solution is prepared by dissolving 0·1 gramme of theobromine in a mixture of 2 c.c. of water and 1 c.c. of hydrochloric acid. Ten c.c. of bromine water are added, the beaker and its contents weighed, the excess of bromine expelled by heat, which is continued until the liquid becomes practically colorless, and the original weight made up with distilled water. This solution when cold stains the skin red. On adding to 2 c.c. of the liquid 1 drop of a 5 per cent. solution of ferrous sulphate and 2 or 3 drops of ammonia solution, an intense indigo-blue coloration is obtained. (A reaction also given by caffeine.)

3. A solution of 0·1 gramme of theobromine in a mixture of 2 c.c. of water and 1 c.c. of hydrochloric acid, mixed with 10 c.c. of decinormal iodine solution (I, 12·7 grammes; KI, 20 grammes; water, 1 litre), gives a dense black precipitate. This is separated by decantation, and dissolved in a 10 per cent. aqueous solution of potassium iodide at 80° C. On cooling, greenish-black crystalline needles of theobromine tetra-iodide are deposited.

Theobromine should leave no residue on ignition, and should melt at 338° to 340° C. The determination of its solubility in alcohol should detect caffeine and other organic substances (alkaloids, glucosides, etc.). Ten c.c. of 95 per cent. alcohol, saturated with theobromine at 21° C., leaves on evaporation a residue of 0·0045 gramme, whilst 10 c.c. of the same alcohol, saturated with caffeine, leaves 0·0930 gramme. Hence theobromine, containing 5 per cent. of caffeine, treated with alcohol, kept at 21° C. for forty-eight hours would give a solution containing in 10 c.c. 0·0290 gramme instead of 0·0045 gramme. C. A. M.

The Assay of Belladonna Plasters. C. E. Smith. (*Amer. Journ. Pharm.*, 1898, lxx., 182-189.)—The belladonna plasters of the American market are nearly always prepared with a base containing rubber and various resins. In their preparation the rhizome of the *Scopolia carniolica* is often used instead of the *Atropa belladonna*, but as the alkaloids are practically the same in both plants, and are present in more uniform quantity in the former, the author considers that there is some excuse for the substitution.

The method of assay which he has found the most accurate and reliable is as follows:

If the sample is of about the strength of the U.S.P. standard, one plaster (about 8 grammes) is taken, or if weaker, proportionately more. The part of the cloth on which the mass is spread is weighed, cut into strips, and stirred up with 50 c.c. of chloroform and 10 drops of 10 per cent. ammonia-water, until the mass is completely dissolved off the cloth. The chloroform mixture is decanted, the rubber precipitated by the addition of 40 c.c. of 91 per cent. alcohol, and the supernatant liquid decanted into a separatory funnel. The cloth left in the first beaker is washed with 25 c.c. of chloroform and 5 drops of ammonia-water, and the washings decanted into the second beaker, where they redissolve the precipitated rubber. The solution is well stirred, the rubber again precipitated by the addition of 20 c.c. of alcohol, and the supernatant liquid added to that in the separatory funnel. This process is repeated until the whole of the mass is removed from the cloth and the beaker.

The combined chloroform-alcohol solutions in the separatory funnel are mixed with 20 c.c. of water containing 2 c.c. of hydrochloric or sulphuric acid and shaken gently for five minutes. The chloroform solution is then drawn off into another funnel, together with any solid matter that may have collected at the line of contact of the two layers, while the acid solution at the top, which contains most of the alkaloid, is transferred to a third separatory funnel. The extraction of the chloroform-alcohol solution with acidulated water is repeated three or four times, or so long as any considerable amount of solid matter collects at the juncture of the liquids.

The combined acid solutions are made alkaline with ammonia, and the alkaloid gently shaken out with successive portions of 20, 10, and 10 c.c. of chloroform. (Violent agitation produces an emulsion.) The combined chloroform extracts are washed with water and transferred to a flask, where the chloroform is evaporated on a water-bath.

The alkaloidal residue is shaken with 4 c.c. of N/20 acid until all the alkaloid is dissolved. Fifty c.c. of water are then added, followed by 0·5 c.c. of a 1 per cent. alcohol solution of hæmatoxylin, and N/20 alkali is run in until the colour of the liquid changes to dull red. The number of c.c. used is subtracted from 4, and the remainder multiplied by 0·0145 (the N/20 factor for atropine and its isomers), and by 100, and the product divided by the net weight.

For example, a plaster weighing 12·25 grammes with the cloth, and the cloth weighing 3·55 grammes, required 1·58 c.c. of N/20 alkali to neutralize the excess of acid.

$$\frac{(4 - 1{\cdot}58) \times 0{\cdot}0145 \times 100}{12{\cdot}25 - 3{\cdot}55} = 0{\cdot}404 \text{ per cent. of alkaloid.}$$

The results of the analyses of eleven samples comprising the products of six manufacturers, including all the prominent American makers, were:

	Per cent. of Alkaloid in Mass.	Weight of Mass in one Plaster. Grammes.
1.	0·571 to 0·594	8·55
2.	0·403 ,, 0·416	8·7
3.	0·509 0·497	—
4.	0·112 0·108	8·65
5.	0·103 0·110	—
6.	0·060 0·058	8·2
7.	0·084 0·081	7·35
8.	0·125 0·116	3·25
9.	0·098 0·101	8·35
10.	0·042 0·047	5·7
11.	0·093 0.096	—

All the samples responded to Vitali's test for solanaceous alkaloids except No. 11, a foreign sample which contained some interfering impurities. Judging by the presence of chlorophyll, No. 2 and No. 11 were prepared from the leaf extract. Only three samples conformed to the U.S.P. standard of strength, all the remainder being much below it. C. A. M.

ORGANIC ANALYSIS.

A New Reaction of Tertiary Alcohols. G. Deniges. (*Comptes Rend.*, 1898, cxxvi., 1277-1279.)—The test here described is applicable to the detection of all compounds which readily yield hydrocarbons of the ethylene series, but especially to tertiary alcohols. The reagent consists of mercuric oxide 50 grammes, sulphuric acid 200 c.c., and water 1000 c.c. On warming two or three drops of a tertiary alcohol with a few c.c. of this solution, a yellow precipitate is rapidly produced, the tint varying with the particular alcohol tested.

In the case of butylic alcohol the yellow compound, when washed and dried in the dark over sulphuric acid, has a composition corresponding to the formula,

$$\left(SO_4\langle{}^{Hg}_{Hg}\rangle O\right)_3 \cdot C_4H_3,$$

which is also the formula for the substance yielded by dissymetric dimethyl ethylene, the corresponding olefiant hydrocarbon. It is soluble in hydrochloric acid with effervescence yielding butylene and mercuric chloride. On boiling the precipitate in its mother liquid for ten minutes, the colour disappears, and finally mercurous sulphate is produced with oxidation of the butylene residue. The test is very sensitive, and is capable of detecting 0·001 gramme of the alcohol.

Tertiary amylic alcohol mixed with three to four times its volume of the reagent and gently warmed, gives a yellow precipitate identical with that yielded by the corresponding ethenic hydrocarbon. On boiling, it is immediately decomposed, giving mercurous sulphate, which is rapidly reduced to metallic mercury.

There is a characteristic difference in the behaviour of pental and tertiary amylic alcohol with the reagent. The former gives an immediate yellow coloration in the cold, followed by the gradual formation of a precipitate of which there is an abundant deposit after ten minutes. Tertiary amylic alcohol scarcely shows any alteration in the cold, though on warming it gives the three stages of the reaction described above. When a drop of the liquid under examination is added to 2 c.c. of the boiling reagent a yellow precipitate is obtained which does not alter on standing in the case of pental, whereas with tertiary amylic alcohol the precipitate becomes white and crystalline and finally gray.

The other tertiary alcohols give analogous compounds with the reagent, always provided they are capable of yielding hydrocarbons of the ethylene series on dehydration. Primary and secondary alcohols do not form these compounds. Isopropylic alcohol, however, which is readily dehydrated with the formation of propylene, gives a precipitate with mercuric sulphate, but with much less readiness than the tertiary alcohols. The esters of tertiary alcohols also give a yellow precipitate with the reagent, and the test is very characteristic in the case of the tertiary amylic nitrite employed in medicine. C. A. M.

Preparation of Soluble Starch. A. Wroblewski. (*Chem. Zeit.*, 1898, xxii., 375.)—20 grammes of rice starch are rubbed down with 100 c.c. of cold water, poured into a 2-litre flask, 1 litre of a boiling 0·5 per cent. solution of caustic potash added quickly, and the whole boiled for 1½ or 2 hours under a reflux condenser till

the liquid becomes thin and pale yellow. The solution is filtered, neutralized with dilute acetic acid (to remove the free alkali and so facilitate the subsequent washing), and precipitated with an equal volume of 95 per cent. alcohol. The starch is filtered off, washed successively with 50 per cent., 95 per cent., and 100 per cent. alcohol, then with ether, and finally dried *in vacuo*. The substance thus obtained is a snow-white powder containing 0·4 to 0·6 per cent. of ash, soluble in about 33 parts of water, and insoluble in cold 35 per cent. or hot 45 per cent. spirit; it does not reduce Fehling's solution, and gives a pure blue colour with iodine. If a very pure material is required, the first product may be dissolved in water and precipitated with alcohol three or four times; it then retains but 0·15 or 0·18 per cent. of ash, while the yield is about 70 per cent.

The solubility of soluble starch in dilute spirit is largely dependent upon the amount of inorganic salts present; and in the same manner with dextrin, etc., if the liquids are too pure, it may be necessary to introduce a small quantity of potassium acetate before precipitation can be effected. F. H. L.

The Solubility of Pentosans in the Reagents employed in the Estimation of Starch. W. H. Krug and H. W. Wiley. (*Jour. Amer. Chem. Soc.*, 1898, xx., 266-268.)—In the determination of starch in a cereal or fodder by digesting the substance at a high pressure with water containing some organic acid, the action of the latter on the pentosans and hemi-celluloses results in the formation of reducing substances. The authors have determined the amount of the pentosans dissolved in this way with the following results:

1. Solubility of the pentosans by digesting for two and a half hours at three and a half atmospheres with 30 c.c. of water and 25 c.c. of 1 per cent. lactic acid.

Substance.	Per Cent. of Pentosans in Substance.	Pentosans Dissolved. In Per Cent. of Substance.	Pentosans Dissolved. In Per Cent. of Pentosans.
Wheat No. 1	5·80	4·63	79·83
Wheat No. 2	5·17	4·66	90·13

2. Solubility of pentosans by digesting for two and a half hours at three and a half atmospheres, with 55 c.c. of water and 0·5 gramme of salicylic acid.

Substance.	Per Cent. of Pentosans in Substance.	Pentosans Dissolved. In Per Cent. of Substance.	Pentosans Dissolved. In Per Cent. of Pentosans.
Wheat No. 1	5·80	4·54	78·27
Wheat No. 2	5·17	4·08	78·91

J. König (*Landwirthsch. Ver. Stat.*, xlviii., 81) states that diastase also acts as a solvent for pentosans and hemicelluloses, and gives figures to show that from 15 to 44 per cent. of the total amount of those substances present pass into solution.

The authors have made a number of starch determinations in various cereals by means of malt-extract and taka-diastase solution, from the results of which they conclude that diastase has no solvent action on the pentosans, and that this method of determining starch is an exact and reliable one. C. A. M.

A Comparison of the Standard Methods for the Estimation of Starch. H. W. Wiley and W. H. Krug. (*Journ. Amer. Chem. Soc.*, 1898, xx., 258-266.)—Referring to Stone's modified method (ANALYST, xx., 19), the authors show that a digestion of the starch with diastase for 2½ hours at 50° C., instead of for one hour at 60° C., gives a result about 1 per cent. higher. Further, by grinding the sample to a much finer powder than Stone prescribes, a considerably higher result is obtained. The comparative mean results obtained with a sample of wheat, analysed by (1) this method, (2) by Reinke's method (digestion under pressure with water containing lactic acid, to prevent the decomposition of sugars formed in the hydrolysis), and (3) by a method differing from Reinke's, by the substitution of salicylic acid for lactic acid were (1) 62·46, (2) 62·32, and (3) 63·09 per cent. In another paper the authors show that by the two latter methods considerable quantities of the pentosans present are also dissolved (*cf.* preceding Abstract). Of the two, the salicylic acid is preferable, since much less caramelization takes place. When it is employed, the amount of starch may be very approximately determined by diminishing the apparent percentage by 1·25. If greater exactness is required, the pentosans should be determined and the percentage divided by three and subtracted from the apparent percentage of starch.

From the results of experiments with the various polarimetric methods, the authors conclude that none of them can be relied upon to give accurate results.

They find Lindet's method (solution of the proteid envelope of the starch cell by means of pepsin and weighing the starch directly, ANALYST, xxii., 20) is more tedious than the common processes, and not so accurate. Small particles of substances other than starch pass through the sieve cloth and are weighed with the starch. On the other hand, a portion of the starch remains attached to the filter, and these two errors may often compensate one another, and an approximately correct result be obtained. The following are some of the figures given in illustration of this :

	LINDET METHOD.			DETERMINATION AFTER CONVERSION INTO DEXTROSE.			
CEREAL.	Starch per cent.	Residue per cent.	Total per cent.	Starch per cent.	Residue per cent.	Total per cent.	Fibre in Starch per cent.
Oats	44·31	0·00	44·31	43·73	0·00	43·73	0·58
Barley	71·41	0·97	72·38	66·47	0·97	67·44	4·94
Rye	61·24	0·79	62·03	58·18	0·79	58·97	3·06
Wheat	66 05	0·60	66·65	60·11	0·60	60·71	5·94

The percentage of starch in the same cereals as determined by the salicylic acid method and the diastase method were :

	Salicylic Acid Method.	Diastase Method.
Oats	46·78	45·05
Barley	68·38	67·56
Rye	62·64	61·55
Wheat	64·41	63·17

A combination of the Lindet method with the salicylic acid was tried with the object of securing a more thorough solution of the starch. Three grammes of the cereal were digested for fourteen hours at 45° C., with 30 c.c. of pepsin solution, the hydrochloric acid neutralized with sodium carbonate, and the substance heated

in the autoclave at 3½ atmospheres, after the addition of 0·5 gramme of salicylic acid. The percentages of starch obtained showed a close agreement with those given by the salicylic acid method alone. The mean results from the same cereals as used in the other experiments were:

Oats, 47·26; Rye, 62·35; Wheat, 64·48.

The combination of the Lindet method with the diastase method is said to have given most satisfactory results, but this part of the subject is still being investigated. In general, the use of pepsin is recommended as a safeguard to ensure the starch completely dissolving.

The diastase method without pressure is very satisfactory with the precautions referred to above. The materials should be ground to the finest possible powder, and extracted with ether to remove the fat. The treatment with diastase should always be repeated after boiling and cooling to about 50° C. (the temperature at which, according to Lintner, the enzyme is most active), and the residue should not show any starch granules when stained with iodine and examined under the microscope. The use of taka-diastase presents many advantages, as it can readily be obtained free from reducing sugar.

The small amount of matter unaccounted for in a complete analysis of cereals is regarded by the authors as probably belonging to the complex class of carbohydrates known as pentosan-ligno-celluloses, but they consider that the quantity is only very minute in cereals, though probably much larger in stalks and straw, and other substances containing a large excess of ligno-cellulose compounds.

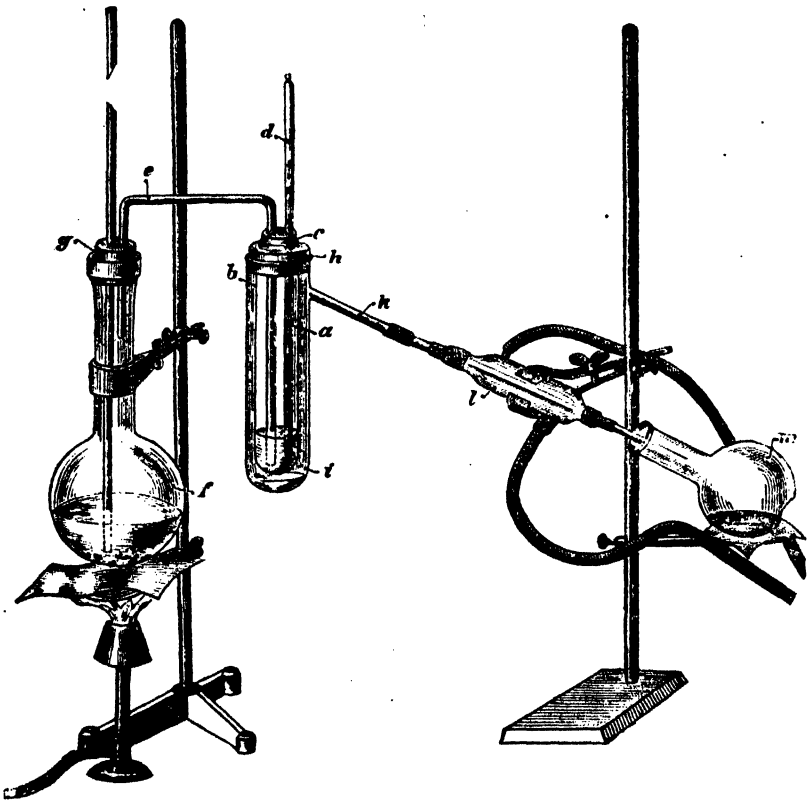
C A. M.

Estimation of Urea. G. Meillère. (*Repert. Pharm.*, 1898, [3], x., 59; through *Chem. Zeit. Rep.*, 1898, 96.)—Sodium hypobromite solution being very unstable, the author prefers to employ the reagent in a nascent condition, preparing it in the nitrometer from a bromide and strong sodium hypochlorite solution, which evolves 30 volumes of active chlorine. 10 c.c. of the urine are diluted with 40 c.c. of water and mixed with 5 c.c. of sodium hypochlorite solution, 5 c.c. of 10 per cent. caustic soda, and 1 c.c. of 20 per cent. potassium bromide. Owing to the dilution of the liquids, warming is scarcely necessary, and a reading can be made in five minutes.

F. H. L.

A New Process for Determining Molecular Weights by the Boiling-Point Method. W. Landsberger. (*Berichte*, 1898, xxxi., 458-473.)—The apparatus employed in this process consists of a test-tube, *a*, in the side of which near the top is a perforation, *b*. The tube is closed by means of a cork, *c*, through which passes a thermometer, *d*, and a conducting-tube, *e*, bent twice at right angles, and is fitted into a larger tube, *i*, connected by means of a side tubulure, *k*, with a condenser, *l*. One end of the conducting-tube passes to the bottom of the inner test-tube, whilst the other is connected with a flask, *f*, in which the pure solvent is heated over a small flame or on a water-bath. The vapour enters the inner tube in which is placed some of the solvent, and in from 2 to 6 minutes a constant temperature is reached, and the boiling-point determined. The inner tube is then emptied, a weighed

quantity of the substance—of which the molecular weight is to be determined—introduced together with some of the solvent, and the vapour again passed in from the flask. The temperature is observed every 8 seconds until 3 consecutive readings in agreement have been obtained. The apparatus is then disconnected, the inner tube, *a*, rapidly withdrawn, the opening *b* and the open end of the tube, *e*, closed with small rubber corks, the tube wiped externally and weighed. It is then washed out with alcohol and ether, dried, and again weighed, the difference between the two weighings giving the weight of solvent and the substance in the tube. The weight of the substance being known, that of the solvent is obtained by subtraction, and the molecular weight calculated by means of the usual formula.



The author gives a table of the results obtained with this apparatus, and these show a close agreement with the figures of other observers. The thermometer employed was graduated in $\frac{1}{20}$ths of a degree. It is stated that a determination can be made within 25 minutes. C. A. M.

INORGANIC ANALYSIS.

Separation of Lead from Copper and Zinc; the Determination of Lead in Presence of Tin, and of Zinc in Bronze, etc. W. E. Garrigues. (*Proc. Eng. Soc., Western Pennsylvania*, 1898, xiv., 80.)—Lead chromate is quite unaffected by ammonia, whereas copper chromate is known to be readily soluble. If, therefore, to a nitric acid solution containing the three metals an excess of potassium bichromate is added,

followed by a decided excess of ammonia, and the liquid heated till the precipitate settles, the lead chromate may be filtered out on a single disc of paper in a Gooch crucible, washed with dilute ammonia, hot water, and alcohol, and finally dried in the water-oven and weighed. Antimony, iron, and probably bismuth interfere with the process.

If 0·5 gramme of a solder containing about 59 per cent. of lead is oxidized with nitric acid, 20 c.c. of strong sulphuric acid added, and the solution evaporated to the point of dense fuming, the meta-stannic acid dissolves completely. On diluting with not more than 80 c.c. of cold water, and stirring for a few minutes, the lead sulphate can be removed by filtration; then by making the filtrate alkaline with ammonia, and again acidifying with sulphuric acid till the liquid reddens methyl-orange, the tin can be recovered, ignited and weighed as stannic oxide. If care be taken not to use too much sulphuric acid, lead and tin may be removed in one operation; in fact, if the copper be thrown down with thiocyanate, the lead, copper, and tin can all be filtered off together, for washing with 3 per cent. sulphuric acid has no effect on either of the two latter precipitates. When small amounts of zinc have to be determined in bronze, the method is unequalled for exactness and speed. F. H. L.

Electrolytic Estimation of Manganese, and the Separation of Iron therefrom. **F. Kaeppel.** (*Zeits. anorg. Chem.*, 1898, xvii., 268.)—By the use of acetone it is possible quantitatively to deposit manganese from its solution as peroxide, to obtain it in the form of a sound coherent layer which can be safely washed even in amounts exceeding 1 gramme in weight, and to weigh it, after drying at 150° or 180° C., as MnO_2 with the certainty that it corresponds exactly to the composition represented by the formula. The original manganese solution, which should contain from 0·3 to 3·0 grammes of anhydrous sulphate, is made up to about 150 c.c., and mixed (proportionately) with 1·5 to 10 grammes of acetone. The liquid is raised to, and strictly maintained at, 50° to 55° C., and electrolysed with a current of 0·7 to 1·2 ampère at a tension of 4 to 4¼ volts for 2 to 5½ hours, the loss by evaporation being constantly made up. To prevent the formation of traces of permanganic acid at the cathode, this electrode should consist of two platinum plates suspended in the basin. The basin is the anode; and when large quantities of peroxide have to be recovered, it may with advantage be roughened with the sand-blast, as recommended by Classen in the case of lead. The deposit is washed without stopping the current (this operation being much simpler than when chrome alum, etc., has been employed), and finally dried for 1 to 2½ hours as aforesaid. Manganese peroxide is, of course, hygroscopic; but if the weighing be done quickly, or repeated after a second interval of drying, the error thus introduced is trivial. The examples quoted show results varying between 36·37 and 36·52 per cent. (mean 36·42 per cent.) in determining 36·42 per cent. of metallic manganese.

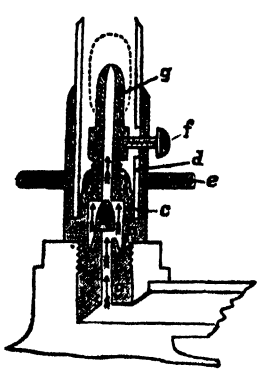
A rough separation of iron and manganese—the former being deposited as metal, and the latter (almost entirely) held in solution as permanganic acid—may be effected as follows; and though it leads constantly to a deficit of 0·3 to 1·0 per cent. in the iron recovered, as it requires no attention, but, proceeding at ordinary temperatures,

may be left going all night, the author thinks it may be found serviceable for some commercial purposes. Moreover, it is the more accurate the larger the proportion of iron in the sample tested. The liquid, which may contain between 0·35 and 1·0 gramme of $FeSO_4 + 7H_2O$ with 0·08 or 0·17 gramme of $MnSO_4$, is poured with constant stirring into a boiling solution of 6 grammes of sodium pyrophosphate; as soon as the whole is clear, 3 or 4 drops of phosphoric acid are added, and, if they produce a turbidity, a little more pyrophosphate. When the liquid is cold, it is electrolysed for 10 to 14 hours with a current of 0·7 to 1·8 ampère and 3·75 to 4·25 volts. The metallic iron adheres firmly to the platinum vessel, which, therefore, need not be given a matt surface; it is washed without stopping the current, rinsed several times in absolute alcohol, and dried at a moderate temperature.

If the electrolysis is conducted at a temperature of 35° to 40° C., the amount of pyrophosphate may be increased to 12 grammes, while the total bulk of the liquid is about 250 c.c. Under these conditions, with a current of 1·8 to 2·5 ampères, the operation is complete in 8 or 9 hours, the quantity of manganese precipitated is very greatly diminished (only a few flakes of oxide are to be seen), and the above-mentioned error in the weight of the iron is practically eliminated. Working on a mixture of about 1 gramme of crystallized ferrous ammonium sulphate with 0·2 to 0·7 gramme of the corresponding manganese salt, the percentage of iron found in the former varied from 14·02 to 14·19 (mean 14·105) per cent. instead of the theoretical 14·28 per cent. Unfortunately, the residual manganese cannot be recovered by electrolytic agency. F. H. L.

APPARATUS.

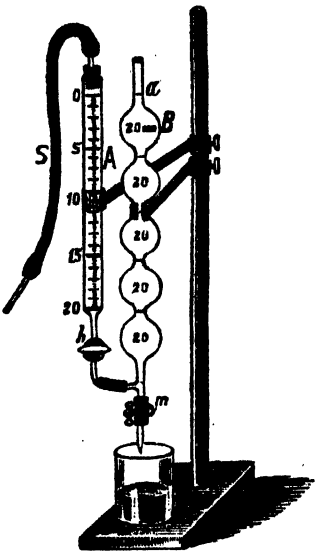
Bunsen Burner with Screw Cock. R. Meyer. (*Chem. Zeit.*, xxii., 226.)—The part *a* in the centre of the foot of the burner is drilled out as shown by the arrows, and carries the cone *b*, which is firmly screwed to it. The burner-tube *d* is screwed to the part *a* at *c*. Over this is passed a tube provided with a disc *e*, and through this tube the screw *f* is passed, and made fast to the burner-nozzle *g*. The lower end of the burner-nozzle screws into the part *a*, so that it can be made to approach or recede from the cone according as it is screwed up or down.



The supply of gas is regulated by turning the disc *e*. When it is turned to the right, the lower end of the nozzle-tube gradually shuts down on to the cone *b*, stopping the supply, the reverse taking place when it is turned to the left. H. H. B. S.

Some Improved Measuring Apparatus. O. Bleier. (*Chem. Zeit.*, 1898, xxii., 298 and 376.)—The apparatus which the author has previously described (ANALYST, xxiii., 55, and 111) is only suitable for the measurement of a pre-determined volume of liquid, and does not lend itself to the process of titration; the following modification,

however, overcomes this disadvantage. A burette (A), graduated in the usual manner, and holding altogether x c.c., is joined base to base with a 5-bulbed tube (B), such as already figured, except that each graduated portion is of the same size, and also holds x c.c. If desired, a second tube (C) of similar construction may be added, in which every division should contain $5x$ c.c. Each vessel preferably carries its own stopcock, while the exit tubes are united into one common orifice closed with a clip. By suitable manipulation of the stopcocks, the bulk of the liquid (corresponding to a certain number of the graduated spaces) can be run out of B and (or) C; and the residue, which requires measuring exactly, out of the burette. Or, if only the latter is provided with a cock, the whole of the liquid can be drawn from B while the cock at the bottom of A is shut, and then by opening the cock, inserting a cork and rubber tube in the top of A and blowing in or withdrawing air, the liquid in B can be brought exactly to the level of one of the marks, and the total volume calculated from the space emptied both in A and B. F. H. L.



A Safety Pipette with Caoutchouc Tube Attachment. **K. Zulkowski.** (*Chem. Zeit.*, xxii., 226.)—The usual form of pipette, whether graduated or constructed to deliver definite quantities, has many disadvantages. When the liquid is sucked up by the mouth, the eye is in an unfavourable position to observe the mark on the instrument and the level of the liquid, and consequently its use is not always altogether free from danger. In the case of large pipettes, the suction has often to be carried out intermittently, and the pipette cannot then be closed with the finger so quickly as could be wished. The adjustment is also often difficult, and there is a danger of the liquid becoming contaminated with the moisture from the mouth. The pipette shown in the accompanying sketch, which sufficiently explains itself, is designed to overcome these drawbacks. H. H. B. S.



REVIEWS.

THE ANALYSIS OF FOOD AND DRUGS.—PART I.: MILK AND MILK PRODUCTS. By T. H. PEARMAIN AND C. G. MOOR. (London: Baillière, Tindall and Cox, 1897.)

Considering that samples of milk and butter form the majority of all the samples taken under the Sale of Food and Drugs Act, and further that samples of milk and milk products are examined in large numbers for various purposes, a treatise on the analysis of dairy products must be regarded as a very desirable manual, the more so as the matter is by no means so simple and easy as on first sight it may appear to many an analyst. Proprietors of the back volumes of the ANALYST have the opportunity of instructing themselves very intimately on the subject, by studying the numerous articles on the composition and examination of dairy products contained in that journal; but the very fact that these are very numerous, and that they are scattered over the whole series of the twenty-two volumes of that journal, makes their perusal a difficult and time-taking task. Under these circumstances, one might say that the book under review meets a decided want. In their preface, the authors state that "in order to keep the book within manageable limits, they are compelled to deal briefly with, or sometimes merely to refer to researches, which would be rightly included in a larger work." Keeping this in mind, it is somewhat surprising to find so many pages devoted to remarks on practical dairying which might readily have been omitted, since many of them are inaccurate; indeed, we find in the book generally a deficiency in correctness of statement and exactitude of expression. Some of the obsolete methods of analysis might well have been omitted, and other processes described in greater detail; while the list of methods actually in use at the present time is not complete. The question of standards and limits is treated at length, without, however, advancing its solution. The chapter on the bacteriology of milk is too short to be exhaustive, and too long for merely touching upon this matter; it contains an account of some of the work lately done in this field of research. The best parts of the book are those treating of the detection of preservatives, and that on condensed milk. If the book, on reaching a second edition, which we hope it will do, is judiciously revised and corrected, it will become a valuable companion, not only to the analyst, but also to the better-educated dairyman. P. V.

APPLIED BACTERIOLOGY. By T. H. PEARMAIN AND C. G. MOOR. Second Edition. (London: Baillière, Tindall and Cox.) Price 12s. 6d.

This work, which we had the opportunity of favourably reviewing some eighteen months since (ANALYST, vol. xxii., 28), now appears in a second edition, and the fact that this is required in a little over twelve months from the publication of the original edition would appear to indicate that the book has fulfilled its mission. The new work is considerably enlarged, there being 457 pages of reading matter against 354 in the old; the arrangement of the several articles has been improved, and the whole work thoroughly revised and brought up to date. References to foreign publications are inserted for the first time. A new chapter on the bacteriology of sewage has been added, and also a new plate illustrating Mr. Stoddart's "Halo" cultures of the typhoid bacterium and its congeners. These additions and improvements cannot fail to render this useful book still more serviceable to all those who are concerned either in the study or in the practical application of bacteriology. W. J. S.

THE ANALYST.

SEPTEMBER, 1898.

OBITUARY.

THE LATE JOHN A. R. NEWLANDS.

It is with very deep regret that we have to record the death, on July 29, at Clapton, of one of our most respected vice-presidents, Mr. J. A. R. Newlands, who was in the sixty-first year of his age.

Mr. Newlands' memory will always be associated with the discovery of the "Periodic Law," which was afterwards worked out more fully and boldly by Mendeléef, Lothar Meyer, and De Chancourtois. Mr. Newlands' first communication on that subject was made to the Chemical Society in 1863 in the form of a note, and in 1866 he presented to that Society a paper entitled "The Law of Octaves, and the causes of the Numerical Relations amongst the Atomic Weights," in which his ideas were further elaborated. His views were received at that time with so much incredulity and even derision by the Chemical Society that they never found a place in its Journal, the subject being deemed of a too theoretical nature to merit publication. Nearly twenty-five years later, however, in 1887, Newlands' work met with authoritative, though tardy, recognition, for in that year the Royal Society awarded him the Davy Medal "for his discovery of the Periodic Law." In the region of applied chemistry he had for many years past devoted his energies mainly, in connection with his well-known brother, Mr. B. E. R. Newlands, to questions connected with the sugar industry.

Mr. Newlands, whose ancestors on the father's side were Scotch, and on the mother's Italian, was of a genial and enthusiastic nature; he was an earnest supporter of reforms in his own country, and even went so far as to serve as an English volunteer under Garibaldi in the war for the liberation of Italy.

We feel that we have lost in Mr. Newlands a remarkable and original man, and one whose affable and kindly disposition had endeared him to the hearts of all who knew him.

PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS.

ON CHICORY, AND VARIATIONS IN ITS COMPOSITION.

By Bernard Dyer, D.Sc.

(*Read at the Meeting, June* 1, 1898.)

Discrepancies between analysts in estimating the proportion of chicory in mixtures of chicory and coffee appear to be unfortunately frequent, and these may be attributed to varying assumptions as to the composition of the chicory present. As there is no satisfactory mode of separating coffee and chicory, the analyst is driven to base his calculations mainly upon the proportion of soluble matter in the sample, and on the assumed average proportions of soluble matter in coffee and chicory. Most analysts experimentally deduce their estimate of soluble matter, and hence the proportions of coffee and chicory, from the density of 10 per cent. infusion of the mixture, and a great many data are on record of the densities of such infusions of coffee and of chicory, but most of them are old. Some analysts—E. W. T. Jones, for example—prefer to evaporate a measured portion of the watery extract and actually weigh the soluble residue. Alfred Smetham in 1883 (Analyst, vii., 73) suggested thoroughly boiling out the sample with water, washing, drying, and weighing the insoluble residue on a counterpoised filter. It presumably makes but little difference which method is used, provided that the same mode of working is adopted for comparative purposes; but, as since the publication of Mr. Smetham's paper, I have myself always determined the insoluble matter directly, and the soluble matter by difference, it is in this form that I now propose to record some results of a recent investigation into the composition of the chicory at present in the market.

Mr. Bannister, in his evidence before the late Select Committee on Food Products Adulteration, in reply to a question from Sir Charles Cameron, endorsed an experience, attributed to Mr. Hehner, to the effect that the soluble matter in chicory appeared to have increased during the last ten or fifteen years, a fact which Mr. Bannister attributed to "difference of growth." My own experience confirms that of Mr. Hehner and Mr. Bannister, but I think there is no reason to suppose that the difference is due to methods of growth. It is more easily and probably accounted for by a custom of less highly roasting.

In 1883 I examined six samples of chicory obtained from various sources, with a view to their being representative of the chicory then in the market, and found that, calculated on the samples dried at 212° F., the matter insoluble in water, after exhaustively boiling and washing, weighed on the average 33 per cent., the variations being from a little over 30 to a little over 36. In 1895 I had occasion to examine a sample of chicory stated to be representative of that which was in use by a very large firm of wholesale grocers, and found only 22 per cent. of insoluble matter in the dried sample.

During the present year I took steps to obtain from several thoroughly representative wholesale houses samples of the chicory in current use, in all cases requesting to be supplied with samples representing any different grades of roasting or colour

that might be in current use. It may be stated that the percentage of moisture varied in extreme cases from 1 per cent. to 4 per cent., but the following results are all calculated on the dry substance. They show the total matter insoluble in water (inclusive of mineral matters), the ether extract, the nitrogen, the total ash, the ash soluble in water, and the sand.

	Total Matter Insoluble in Water.	Ether Extract.	Nitrogen.	Total Ash.	Ash Soluble in Water.	Sand.
	Per cent.	Per cent.	Per cent.	Per cent.	Per cent.	Per cent.
Chicory "nibs," described as "medium roast" ...	22·40.	2·57	1·53	4 63	2·50	0·70
Chicory "nibs," described as "dark roast" ...	50·30	2·43	1·67	4·70	2 99	0·30
Ground chicory	22·27	2·17	1·33	5·53	2·43	1·43
,, ,, ...	21·50	1·90	1·34	5·23	2·07	1·43
,, ,, ...	35·50	3·43	1·50	5·13	2·57	0·77
,, ,, ...	37·80	3·87	1·52	8·23	1·60	3·97
,, ,, ...	22·77	3·17	1·25	5·13	3·30	1·60
,, ,, ...	22·50	3·67	1·23	5·73	3·23	1·63
,, ,, ...	23·50	2·60	1·29	5·63	2·97	1·47
,, ,, ...	22·50	2·60	1·29	5·33	3·20	1·47
,, ,, ...	22·63	2·57	1·29	5·70	2·60	1·47

It will be seen that in eight out of the eleven samples the matter insoluble in water ranged from 21·50 to 23·50 per cent. One sample contained 35·50 per cent., one 37·80, and one 50·30 per cent. of insoluble matter.

I have myself made no experiments to ascertain what percentage of insoluble matter is indirectly indicated by the figures usually assumed to represent the specific gravity of a 10 per cent. infusion of chicory, but I believe they may be taken as roughly corresponding to about 70 per cent. of soluble matter and 30 per cent. of insoluble matter, corresponding to the 70 per cent. of soluble matter assumed by E. W. T. Jones as representing chicory in his direct method of determination.

There is no doubt that, if the samples I have recently obtained fairly represent the chicory at present in the market—and I have every reason to think they do—any estimates deduced from density determinations with the aid of the older data would result in too high an estimate of the proportion of chicory, since they assume a much less proportion of soluble matter than that which is present in the greater part of the chicory at present in use. It is quite true, on the other hand, that with some kinds of chicory at present in use, reliance on the old figures might cause an error on the side of considerably underestimating the proportion of chicory; but an underestimate of the inferior article is, from the public analyst's point of view, obviously a matter of far less moment than an overestimate, which would reflect unjustly upon a seller of the article. It is indeed remarkable that, after attention had been drawn to the matter, the old figures should have still remained in use.

The following experiments were made with the sample marked "chicory nibs, medium roast," which gave originally 22·40 per cent. of insoluble matter. A portion

of the sample, after grinding and drying, was placed in a platinum capsule over a very low argand flame, and gently heated with thorough stirring until it had lost about 5 per cent. of its weight. A similar portion was similarly treated until it had lost about 10 per cent. of its weight. Calculating on the original weight taken, the proportion of insoluble matter in the first experiment rose from its original figure of 22·40 per cent. to 28·50 per cent., and during the second experiment it rose to 41·50 per cent. Seeing that there was a loss in the two experiments of 5 and 10 per cent. respectively, the percentage of insoluble matter in the actual roasted chicories would be 30 per cent. as the result of the first experiment, and 46 per cent. as the result of the second experiment. In neither case was there any appearance which could be described as that of burning. It seems clear, therefore, that the percentage of insoluble matter is mainly a function of the mode and duration of roasting.

It may be of moment to mention here that, on exhaustive boiling, Smetham, in 1882, found a number of coffee samples to contain about 75 per cent. of matter insoluble in water. Three samples of coffee of known purity which I examined in 1883 by this method gave from 72 to 73·3 per cent., averaging 72·9. In 1895 I made determinations in a batch of 16 samples of pure coffee, which showed a range of from 69·4 to 74·3 per cent., averaging 72 per cent. In 1897, 27 pure samples ranged from 71·8 to 75·9 per cent., averaging 73·7 per cent. I have found as little as somewhat over 68 per cent. of insoluble matter (on the dry sample) in a ground coffee in which no chicory was discernible, and which on all other grounds appeared to be genuine; but it is quite possible that in this, as in some other cases somewhat approximating to it, sugar may have been used in roasting the coffee, though no evidence was obtainable that such was the case.

Discussion.

Mr. Cassal said that quite five years previously he had found as much as 79 per cent. of soluble matter in chicory, and had in consequence been in the habit of depending upon the number 79, which almost exactly corresponded with the minimum figure now given by Dr. Dyer for insoluble matter, viz., 21·5 per cent. With regard to the samples yielding 50·3 and 37·8 per cent. of insoluble matter, it was evident that such cases were altogether abnormal, and that what could properly be regarded as "chicory" was not being dealt with. In a substance like chicory, continued roasting necessarily increased the proportion of insoluble matter.

Mr. Chattaway inquired whether the tinctorial power increased as the roasting was carried to a higher degree. A highly caramelized sample might be expected to yield a darker solution than one that has been less highly roasted.

Mr. Chapman observed that the relative proportions of soluble and insoluble matters would depend to some extent, not only upon the actual temperature, but also upon the conditions under which the roasting process was carried on. In roasting a substance like chicory, containing all kinds of caramelizable bodies, soluble compounds would be formed up to a certain point, after which such soluble compounds would to some extent be decomposed, with the result that the soluble matter, after reaching a maximum, would diminish again. In the manufacture of caramel from sugar, if the heating were effected too quickly, or the caramelized sugar poured into water at

too early a stage in the process, the maximum coloring power and soluble extract would not be obtained. It was, he thought, to be expected that considerable variations from what might be called the normal extract should from time to time be observed.

Mr. CASSAL added that he thought the figure usually taken for soluble matter in pure coffee, viz., 24 per cent., was sometimes too easily accepted. He had himself found as much as 29 per cent. of soluble matter in genuine coffee.

Dr. DYER said that he had once found 68 per cent. of soluble matter in a sample of coffee that was undoubtedly free from chicory. The sample, however, might have been roasted with sugar, and this was difficult to detect.

Dr. VOELCKER asked how much oil chicory usually contained.

Dr. DYER said that the oil was from 2 to 3½ per cent., but it did not seem to have any relation to the other constituents. In the sample containing 50 per cent. of insoluble matter the oil was 2½ per cent.; in the two lowest samples it was 2½ and 3½ per cent. respectively. The highly roasted samples were somewhat darker than the others, but in the case of a mixture the colour of the coffee itself would also be liable to variation.

ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

FOODS AND DRUGS ANALYSIS.

Detection of Annatto and other Coloring Agents in Milk. J. Froidevaux. (*Ann. Chim. Analyt.*, vol. iii. [4], pp. 110-114.)—Of the various coloring agents added to milk, annatto and *orange III. Poirier* impart a flesh tint, saffron a slightly orange yellow, carrot an orange-yellow, and turmeric a slightly greenish-yellow tinge to the liquid. The last-named may be detected by the change to brown-yellow ensuing on the addition of ammonia. Hydrochloric acid (5 per cent. by volume) deepens the shade due to vegetable coloring matters and produces a clear rose coloration in presence of *orange III.*

The detection of these colorants is facilitated by coagulating the casein with a little rennet at 25° to 30° C. during fifteen hours, the colour lakes thus formed being more pronounced in shade—except in the case of *orange III.*—than when in solution.

Saffron and turmeric will impart a yellow stain to white filter-paper immersed in the whey for four days, the former colorant being distinguishable by the yellow stain—turning to slightly orange-yellow in presence of very dilute (*e.g.*, 2 per cent.) sulphuric acid—which it gives to filter-paper on immersion for four days in the liquid left after treating the milk with an equal volume of Adam's reagent,* and removing the

* This reagent, which is employed by Adam in his process for estimating fat in milk, consists of:

Alcohol (specific gravity 0·96°)	833 c.c.
Ammonia (specific gravity 0·925)	30 c.c.

These are made up to a litre with distilled water, and then 1100 c.c. of ether washed with water added.

upper ethereal layer of dissolved fat. Under the same treatment annatto gives an orange-red, changing to blue under the influence of 1 drop of strong sulphuric acid, and a bright rose colour in presence of a 2 per cent. solution of acid. C. S.

Detection of Annatto in Milk. A. Leys. (*Ann. Chim. Analyt.*, vol. iii. [5], pp. 149-151. See preceding abstract.)—The presence of this substance in milk can be detected more rapidly than by the method proposed by Froidevaux, if, after treating the suspected liquid with Adam's reagent and removing the supernatant ethereal solution, the casein in the lower layer be precipitated by the gradual addition of 50 per cent. of a 10 per cent. solution of sodium sulphate, without agitation. This treatment leaves the coloring matter in solution, from which it may be extracted by agitation with colorless amyl alcohol, the emulsion thus formed being dissipated by slowly heating the mixture to 80° C. The reddish alcoholic solution is evaporated to dryness and redissolved in hot, slightly alcoholized ammoniacal water, a strip of bleached cotton-cloth being immersed in the liquid, which is then concentrated on the water-bath. The cotton absorbs the coloring matter, and, after being lightly washed, is immersed in a weak acid (*e.g.*, citric acid), which in the case of annatto immediately changes the colour to a clear rose. This reaction does not ensue in the case of saffron, turmeric, etc., or in uncolored milk, notwithstanding that the cotton is dyed. C. S.

New Reactions for the Detection of Aldehydes in Alcohols. C. Istrati. (*Bulletinul Societatii de Sciinte, Bucuresci*, vol. vii. [2], pp. 163-170.)—The method adopted by Barbot and Jandrier (ANALYST, xxi., 295) can be improved by taking *exactly* 2 c.c. of the impure alcohol, adding thereto 0·2 c.c. of a saturated alcoholic solution of the reagent, and, after mixing the two, running in *exactly* 1 c.c. of sulphuric acid from a pipette, the tube being held at an angle of 45°.

The coloration produced at the moment of contact is noted, as is also the change after a lapse of half an hour. After partial agitation, to increase the surface of contact, the coloration is again noted at the end of half an hour, and once again half an hour after complete agitation. The liquid is then mixed with 10 c.c. of water, and examined half an hour and twenty-four hours later, the latter observation being extremely useful, as any precipitate formed will have had sufficient time to settle down.

Greater uniformity of conditions is ensured by the use of the alcoholic solution of the reagent, but such solution, being unstable, must be freshly prepared. In the case of liquid reagents, such as guaiacol, 1 drop is used instead of 0·2 c.c.

The reactions given by ten other substances than those examined by Barbot and Jandrier are tabulated below, those printed in italics referring to dilutions of $\frac{1}{10,000}$, and those in thick type to dilutions of $\frac{1}{100,000}$. The remainder refer to $\frac{1}{1,000}$ strength :

	Methanal.	Ethanal.	Valeral.	Acrolein.	Furfural.	Benzaldehyde.	Methylal.	Acetal.
Pyrogallol	—	Reddish-yellow	*Orange-red*	Brown-red	**Brown-yellow**, turning to black	*Brown-red*	Carmine-red	Orange-yellow
β-naphthol	Orange-yellow, with green fluorescence; *red* pp. with water	**Yellow**	*Yellow*	Golden-yellow	*Carmine-red*	**Crimson**	Orange-red; strawberry pp. with water	—
α-naphthol	—	*Reddish-yellow*	Orange-red	—	Red, nearly black	*Idem.*	Yellow, red above, green below with water	Orange-yellow
Phenylhydrazine	Light brownish-green	—	—	—	**Green**, more characteristic than the others	Brownish-red, better than with β	—	—
Hydroquinone	Reddish-yellow	Yellow	Golden-yellow	—	—	—	—	—
Guaiacol	—	Rose-yellow	—	Reddish	—	—	Violet-red	—
Orcine	—	Brown-yellow	—	—	**Brown-yellow**	—	—	—
Vanillin	—	Blackish-green; grey-blue pp. with water	—	—	—	Brown-red, changing to green	—	Blackish-green; blue pp. with water
Morphine	Violet	—	—	—	—	—	Heliotrope-violet	—
Codeine	Violet-yellow	—	—	*Green* (1 drop of reagent)	*Brownish-yellow* (slow)	—	—	—
Thymol	—	—	—	—	—	Orange-yellow	—	—
β-orthonaphthoic acid	Brown-yellow?	—	*Yellow* (slow)	Golden-yellow, green fluorescence	**Brown-yellow**, turning to black	—	—	—
Resorcin	—	—	—	—	—	*Yellow*	—	—
Protogallic acid	Greenish-yellow, disappearing with water	—	—	—	—	—	Greenish-yellow, disappearing with water	
Phenol	—	—	—	—	Yellow	—	—	—
Phloroglucin	—	—	—	—	*Brown-red*, turning to black	—	—	—
Gallic acid	Greenish-yellow	—	—	—	—	—	—	—
Camphor	Golden-yellow	—	—	—	—	—	—	—
Brucine	—	—	—	—	—	Rose, *brown*, violet (very slow)	—	—

Estimation of the Dry Extract in Wine. **A. Celleri** (*Bull. du Ministère de l'Agriculture*, through *Ann. Chim. Analyt.*, vol. iii. [6], pp. 204-206.)—The period of evaporation may be reduced from the three days required for the Magnier vacuum method (ANALYST, xxi., 66) to seven and a half hours by employing a temperature of 80° C., and a U-tube apparatus with limbs of unequal diameter, the narrow one being connected with a bulb-tube containing sulphuric acid, and the larger one half filled with a piece of fine sponge, and fitted with an effluent tube for the condensation of the evolved vapour. This apparatus is dried by heating for about an hour over the boiling water bath until the difference between two weighings, at intervals of half an hour, does not exceed 1 milligramme. Ten c.c. of wine are then introduced by means of a pipette and absorbed by the sponge, and the tube is heated on the water-bath at 80° C.—a current of air being passed continuously through the apparatus—until found to be perfectly dry below the cork and of constant weight (with a latitude of 1 milligramme at half-hour intervals). The increase in weight represents the amount of dry extract in the wine.

At 80° C. the loss of glycerol by evaporation does not exceed 0·05 per cent., but this temperature should not be exceeded. The results obtained by this method are fairly constant, and are only 0·5 gramme per litre inferior to those yielded by evaporation *in vacuo*. C. S.

Action of Charcoal Decolorizers on Wines. **H. Astruc.** (*Ann. Chim. Analyt.*, vol. iii. [6], pp. 183-191.)—As the result of experiments with ten kinds of charcoal decolorizers, comprising crude and purified bone blacks, lamp blacks, and vegetable charcoal, the author concludes that there is little difference between the crude and purified animal blacks so far as their organoleptic and colorimetric influence on wine is concerned, the purified kinds being, however, slightly preferable. Their decolorizing power is best preserved by storage in a moist condition after washing.

All the decolorizers absorbed a little alcohol (0·4 to 1·5 per cent. out of a total of 7·8); a small proportion of the total acidity; 0·5 to 2·65 per cent. of the glycerol (total, 4·5 per cent.), and 0·95 to 2·65 per cent. out of a total of 3·45 per cent. of tannin, besides extracting coloring matter. The crude bone blacks are distinguished from the purified blacks and vegetable charcoals by removing almost the whole of the tartrates and a larger proportion of glycerol, and especially by increasing the amount of mineral matter in solution to double its original weight, the increase being entirely in insoluble ash-constituents (chiefly lime and phosphoric acid), whereas the soluble portion is diminished.

The decolorizing power of the vegetable blacks is low, and necessitates the use of a twofold or triple quantity, the effect of which on the chemical constitution of the wine is greater than that of a suitable amount of animal black. The flavour of the wine is also more seriously affected than by the latter. C. S.

The Estimation of Oil in Emulsions. **Dr. Schneegans.** (*Journ. Pharm. d'Alsace-Lorraine*, 1897, p. 323; through *Rev. Chim. Analyt. Appl.*, vol. vi. [8], p. 130.)—Three hundred parts of the oily emulsion under examination are evaporated with

50 parts of white of egg and coarse sand, 50 parts of anhydrous sodium sulphate being added and the evaporation completed, with continued stirring to prevent caking. The residue is pulverized and extracted with ether, the fat being recovered by evaporating the solvent in a tared beaker. C. S.

Acid Glycero-phosphates. Adrian and Trillat. (*Journ. Pharm. Chim.*, 1898, vii., 527-532.—The acid salts of glycero-phosphoric acid can be prepared by decomposing a normal glycero-phosphate with sulphuric acid:

$$2PO\begin{array}{l}\diagup O \\ -O \\ \diagdown OC_3H_5(OH)_2\end{array}\!\!>Ba + H_2SO_4 = BaSO_4 + PO\begin{array}{l}\diagup O-Ba-O \\ -OH \\ \diagdown OC_3H_5(OH)_2\end{array}\begin{array}{r} \\ -OP, \\ (OH)_2C_3H_5O\diagup\end{array}$$

or by double decomposition between a soluble sulphate and an acid glycero-phosphate:

$$Ba\left[OPO\begin{array}{l}\diagup OH \\ \diagdown OC_3H_5(OH)_2\end{array}\right]_2 + MgSO_4 = BaSO_4 + Mg\left[OPO\begin{array}{l}\diagup OH \\ \diagdown OC_3H_5(OH)_2\end{array}\right]_2$$

They are employed in the preparation of organic glycero-phosphates, such as those of quinine, cocaine, etc.

The method of determination given by the author is to dissolve from 1 to 2 grammes in 50 c.c. of water previously boiled, and to titrate the filtered solution with normal potash, using phenolphthalein as indicator. The amount of acid salt is then calculated from an equation like the following for the barium salt:

$$Ba\left[OPO\begin{array}{l}\diagup OH \\ \diagdown OC_3H_5(OH)_2\end{array}\right]_2 + 2KOH = H_2O + PO\begin{array}{l}\diagup O \\ \;\; O \\ \diagdown OC_3H_5(OH)_2\end{array}\!\!>Ba + PO\begin{array}{l}\diagup OK \\ -OK \\ \diagdown OC_3H_5(OH)_2\end{array}$$

Cf. Analyst (this vol., 45). C. A. M.

Estimation of Iodoform. G. Meillère. (*Ann. Chim. Analyt.*, vol. iii. [5], pp. 153, 154.)—The difficulties encountered in the estimation of iodoform in presence of organic substances may be overcome by the following method: The iodoform is placed in a flask where it is treated with 25 c.c. of pure nitric acid (free from chlorine), followed by a slight excess of silver nitrate (1·75 grammes per 1 gramme of iodoform), this order being observed in order to prevent the explosion resulting from the contact of iodoform and the silver salt in the dry state. The flask being connected with a Liebig bulb-tube containing silver nitrate solution, the contents are slowly heated, without actually boiling, for ten minutes, after which the temperature is raised to effect the decomposition of the iodoform. When nitrous fumes cease to be evolved, the liquid, after dilution to 150 c.c., is heated until clear, the precipitate is collected on a tared filter and washed, being finally dried at 100° C. Should the nitrate solution in the bulb-tubes become turbid, it must be united to the contents of the flask.

In two determinations made with 1 gramme of iodoform an average of 1·7835 grammes of silver iodide was obtained, the theoretical yield being 1·789 grammes.

C. S.

TOXICOLOGICAL ANALYSIS.

A Strychnine-like Alkaloid found in a Corpse. P. Mecke. (*Pharm. Zeit.*, 1898, xliii., 300; through *Chem. Zeit. Rep.*, 1898, 128.)—During the examination of a partially decomposed body the author met with an alkaloid which chemically might easily be mistaken for strychnine, but which physiologically did not injure frogs, had scarcely a bitter taste, and did not produce the least effect on animals when injected subcutaneously. With picric acid it gave a yellow crystalline precipitate, becoming violet on treatment with sulphuric acid and bichromate. Bichromate alone produced a yellow crystalline precipitate, changing to blue with sulphuric acid. Bichromate and acid together yielded a blue, afterwards a reddish, colour. Ferricyanide gave a yellow precipitate, turned violet by acid. On the other hand, chlorine-water gave (as with strychnine) a milky liquid, but when after evaporation the residue was treated with ammonia it became a dirty green. It dissolved in sulphuric acid to a yellowish liquid, afterwards changing to cherry- and rose-red. Fröhde's reagent dissolved it, giving a dull violet colour, which finally became olive and then green. The substance is not the same as that discovered under similar conditions by Amthor in 1887.

F. H. L.

ORGANIC ANALYSIS.

The Examination of Semi-Woollen Textile Fabrics. S. Kapff. (*M. Text*, 1898, 193. Through *Zeit. angew. Chem.*, 1898, 452, 453).—In the author's opinion all the methods proposed for determining the proportion of cotton and wool in a mixture are inaccurate. He recommends the following process: Five grammes of the sample are extracted with ether for the determination of the fat. The residue is well stirred for thirty minutes in a mixture of 3 parts of hydrochloric acid with 100 parts of water, which is brought to the boiling-point before the introduction of the substance, and the flame subsequently withdrawn. It is then boiled with water for fifteen minutes, washed free from hydrochloric acid, dried for two hours at 100° C., and left exposed to the air for about twelve hours for the fibres to assume their original moisture. The loss in weight gives the amount of weighting material, coloring matter, etc. This treatment also removes certain natural constituents of the wool and cotton, whilst the fibres themselves are attacked, though but slightly.

The residual fibres are separated by heating them to the boiling-point with 250 c.c. of water containing 5 grammes of sodium hydroxide. The cotton which is left is collected on a filter, washed first with a litre of water, then with half a litre of water slightly acidified with hydrochloric acid, and finally with water until free from acid. It is then dried for two or three hours at 100° C., allowed to stand exposed to the air for about twelve hours, and weighed. The addition of 4·5 per cent. to the weight obtained gives the amount of cotton-fibre in the mixture, this correction being based on the results of experiments which showed that pure cotton-wool on treatment with soda as described above loses almost exactly that amount. C. A. M.

Estimation of Nicotine in Tobacco. C. C. Keller. (*Ber. Pharm. Ges.*, 1898, viii., 145; through *Chem. Zeit. Rep.*, 1898, 159.)—Six grammes of dried tobacco are repeatedly agitated during half an hour with 60 grammes of ether, 60 grammes of petroleum spirit, and 10 c.c. of 20 per cent. aqueous caustic potash. After standing three or four hours, 100 grammes of the ethereal liquid (equal to 5 grammes of tobacco) are filtered off, a powerful current of air is driven through it for one or one and a half minutes to remove the free ammonia, 10 c.c. of alcohol, 1 drop of a 1 per cent. solution of eosin B, and 10 c.c. of water are added, and the whole is well shaken. A slight excess of decinormal HCl is then run in, and the liquid is titrated with decinormal ammonia. One c.c. of $\frac{N}{10}$ HCl equals 0·0162 gramme of nicotine.

F. H. L.

Detection of Acetone in Urine. B. Studer. (*Schweitz. Wochenschr. Chem. Pharm.*, 1898, xxxvi., 149; through *Chem. Zeit. Rep.*, 1898, 127.)—The author's process is a combination of the Legal and the Lieben-Dragendorff methods. Fifty c.c. of urine and 5 c.c. of dilute sulphuric acid are distilled in a fractionating flask until about 3 c.c. have passed over. The distillate is mixed with 6 to 10 drops of a freshly-prepared 10 per cent. solution of sodium nitroprusside and 1 to 2 drops of caustic soda to make it alkaline. In the presence of acetone the liquid becomes purple-red; in its absence, yellow or orange. If a pale red is produced, suggesting a trace of acetone, 6 or 8 drops of glacial acetic acid are added, when an intense wine-red colour confirms the test; while, if the liquid turns yellow again, acetone does not exist in the urine.

F. H. L.

Estimation of Uric Acid, based on the Insolubility of Ammonium Urate. J. Triollet and J. Eury. (*Union Pharmaceut.*, May 5, 1898; through *Rev. Chim. Analyt. Appl.*, vol. vi. [10], p. 166.)—The authors recommend the employment of the method proposed by Otto Folin, 100 c.c. of urine being rendered slightly alkaline by agitating with 10 grammes of ammonium sulphate, and the precipitate, collected after two hours' standing, washed with 50 c.c. of a 10 per cent. solution of the same salt. It is then redissolved in (slightly alkaline) boiling water, the solution diluted to 100 c.c. after cooling, and treated with 15 c.c. of sulphuric acid, which causes the temperature to rise to 55° to 60° C. Titration is effected by the Hopkins method with $\frac{1}{20}$ normal potassium permanganate until a persistent rose coloration ensues, 1 milligramme being added to the result as correction for loss due to the solubility of the ammonium urate.

C. S.

Colorimetric Estimation of Aceto-acetic Acid in Diabetic Urine. F. Martz. (*Union Pharmaceut.*, May 15, 1898; through *Rev. Chim. Analyt. Appl.*, vol. vi. [10], pp. 165, 166.)—The reagent employed is ferric chloride, which gives a violet coloration with aceto-acetic acid in urine before, but not after, the latter has been boiled.

In cases where the coloration is due to this acid alone 5 c.c. of officinal ferric chloride are added to 100 c.c. of urine, and the filtered liquid is placed in a test-glass, where it is compared with 20 to 50 c.c. of a solution of 1 gramme of aceto-

acetic acid in 100 c.c. of water, plus 5 c.c. of the reagent, the test-liquid being made up with water until both liquids are of equal depth of colour.

In presence of other substances giving the same reaction two determinations are necessary, one previous to and the other after boiling the urine, the coloration due to the acid in question being calculated from the difference between the two results.

C. S.

Detection of Glucose by means of Litmus. A. M. Julhiard. (*Ann. Chim. Analyt.*, vol. iii. [5], pp. 154, 155.)—Litmus being (like indigo carmine) decolorized by glucose in an alkaline solution, the author recommends the following as a ready test for the detection of that sugar in urine. A little sodium carbonate and a few drops of litmus tincture being added to the suspected urine, the liquid is heated to boiling, whereupon if containing sugar it will assume a dirty yellow colour. The reaction is more apparent after the precipitate has settled down, but it is not permanent, the blue coloration gradually reappearing on exposure to the air. Litmus-paper is also decolorized under the same conditions, and may therefore be conveniently employed instead of litmus solution. C. S.

The Estimation of Succinic Acid in the presence of Tartaric and Lactic Acids. F. Bordas, Joulin, and Raezkowski. (*Journ. Pharm. Chim.*, 1898, vii., 417, 418)—This is based on the difference in solubility of the silver salts of the three acids. On adding a concentrated solution of silver nitrate to a solution of their neutral soluble salts, the succinic acid is precipitated completely, the tartaric acid partially, while the lactic acid remains in solution.

In the determination, the solution containing the three acids is exactly neutralized with decinormal potash, an excess of a concentrated solution of silver nitrate added, and the precipitate filtered off and washed free from silver. Only the silver succinate is left on the filter, for the washing removes the silver tartrate completely.

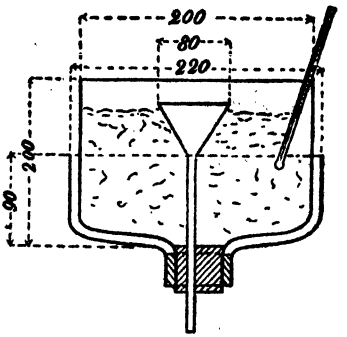
The precipitate is washed into a flask, and two drops of a solution of potassium chromate added, which decompose the silver succinate with the formation of silver chromate. Decinormal sodium chloride is then added, until the precipitate becomes white and the liquid assumes a yellow tint, and finally the excess of sodium chloride is titrated with decinormal silver nitrate.

By subtracting the number of c.c. used in the last titration from the amount of sodium chloride taken, the volume of decinormal silver nitrate required to precipitate the succinic acid present is obtained. One c.c. of the silver nitrate corresponds to 0·0059 gramme of succinic acid. C. A. M.

Quantitative Estimation of Paraffin in Petroleum and Lignite-tar Distillates. D. Holde and L. Allen. (*Mittheil. Kgl. Techn. Versuchsanst.*, 1898, No. 2; through *Chem. Rev. Fett- u. Harz-Ind.*, vol. v. [6], pp. 112-115, [7], pp. 131-134.)—The present investigations were designed to ascertain the suitability of the alcohol-ether method for a wider series of oils than those previously tested therewith

(*Mittheilungen*, 1897, p. 211). The Aisinmann alcohol method was also critically examined.

The apparatus for cold filtration was altered to the form and dimensions shown in the annexed figure and acted well, enabling a constant temperature of −20° to −21° C. to be maintained with one charging long enough for the performance of a complete quantitative determination. The practice of ascertaining beforehand the minimum quantity of alcohol-ether necessary for dissolving the oil, and of effecting the solution of the latter at room temperature, greatly facilitates the subsequent operations and the separation of the paraffin, 0·4 per cent. of which was by this means recovered from a paraffin-oil wherein it was otherwise quantitatively undeterminable.



In applying the method to oils rich in paraffin, from 0·5 to 2 grammes of substance were dissolved in a minimum quantity of sulphuric ether, the solution clarified by the addition of an equal volume of absolute alcohol, and after insertion in the cooling-apparatus and dilution (if necessary) until filterable by the aid of alcohol-ether (1 : 1), the paraffin was separated by filtration. The filtrates were then evaporated and treated as before, in order to recover any residual dissolved paraffin. In all cases the paraffin was dissolved in petroleum spirit (b. p. 50° C.) and carefully re-evaporated, any moisture present being removed by evaporation with a little absolute alcohol.

As regards the solubility of paraffin in absolute alcohol, this was found to be greater than assumed by Aisinmann, 100 parts of solvent taking up 0·10 to 0·19 part at 20° C., and 0·015 to 0·017 part at −15° C. In alcohol-ether the solubility is greater (0·03 to 0·06 per cent. at −19° C. to −20° C.), and in both cases it varies inversely with the melting-point. The presence of oil in the alcohol-ether mixture, however, reduces the solvent power of the latter for paraffin, so that this source of error is counteracted when the method is applied for the estimation of paraffin in mineral oil distillates, the loss by solution ranging from 0·2 per cent. in oils containing 1 to 5 per cent. of hard paraffin, to an average of 1 per cent. in oils richer (30 to 90 per cent.) in that substance. For soft paraffins the results require to be compared with those obtained by treating mixtures of known paraffin content.

The objection urged by Eisenlohr against methods employing a higher temperature than 100° C. for drying the paraffin—on the ground that soft paraffin suffers loss by evaporation under such conditions—was investigated, but found to be baseless so far as the alcohol-ether method is concerned, the abbreviated (quarter of an hour) exposure to 105° C. resulting in a loss so small as to be negligable in technical analyses. On the other hand, a sample of paraffin supplied by Eisenlohr was found to lose considerably by this treatment, and it is therefore proposed in such cases to dispense with the evaporation at 105° C., and merely heat on the water-bath until the odour of the solvent disappears, the drying being completed in the desiccator.

Interesting results were obtained by applying the alcohol-ether method to lignite-

tar distillates, the figures obtained showing a yield of paraffin (m. p. 26° to 53° C.) from 1 to 3 per cent. higher than actually furnished in the refinery; and in one case the accurate characterization of the practical value of the material was shown by the recovery of 5 per cent. of worthless paraffin (m. p. 26° to 27° C.) from a distillate from which none could be separated in the factory. These experiences controvert Eisenlohr's opinion that the method is unsuitable for lignite-tar distillates.

An investigation of Aisinmann's alcohol method showed that accurate results are only obtainable when the paraffin greatly predominates (*e.g.*, 80 per cent.) in the mixture, and even then the alcohol-ether is preferable on account of its greater ease in manipulation. C. S.

The Reactions in Hübl's Method of Iodine Absorption. J. J. A. Wijs. (*Zeit. angew. Chem.*, 1898, 291-297.)—Ephraim (ANALYST, 1895, xx., 176) attributed the additive power of Hübl's solution to the presence of iodine monochloride, and described a method of determining the halogen absorption of fats by means of a solution of that substance. The author finds that the formation of the iodine chloride takes place in accordance with the equation

$$(1)\quad HgCl_2 + 4I = HgI_2 + 2ICl,$$

and considers that the equivalence of these substances is proved by the fact that it is possible to prepare a solution giving the same results as that of Hübl, by dissolving mercuric iodide in an alcoholic solution of iodine chloride.

The iodine chloride thus formed reacts with the water of the alcohol yielding hypoiodous acid and hydrochloric acid

$$(2)\quad ICl + H_2O = HCl + HIO;$$

and this acid is continually being slowly decomposed into iodic acid and free iodine, which substances in the presence of hydrochloric acid in alcoholic solution again produce iodine monochloride, so that a complicated equivalence results.

On adding the potassium iodide and water to the solution before titration, the following reactions take place:

$$(3)\quad ICl + KI = KCl + 2I;$$
$$(4)\quad HCl + HIO + KI = KCl + H_2O + 2I;$$
$$(5)\quad 5HCl + HIO_3 + 5KI = 5KCl + 3H_2O + 6I.$$

Theoretically in a blank determination the whole of the iodine should be found on titration, and the solution should contain no free acid, but in practice this is never the case, for the older the solution the less the iodine titre and the greater the acidity, the deficiency in iodine being equivalent to the acid formed. The author explains the alteration which occurs in the solution on keeping by the fact that hypoiodous acid oxidizes the alcohol to aldehyde.

$$2HIO + C_2H_6O = 2I + H_2O + C_2H_4O;$$

and that this causes a disturbance in the equivalence of the solution. Ordinarily each molecule of hypoiodous acid gives on titration (4) two atoms of iodine, but when there is aldehyde formation only one atom is liberated and the titre is correspondingly smaller. On the other hand, for each molecule of hypoiodous acid reduced a molecule

of hydrochloric acid is set free, which is not neutralized as in equation (4), and which therefore leaves the liquid acid. This hydrochloric acid the author terms "excess acid."

The stability of Hübl's solution may therefore be increased by keeping the concentration of hypoiodous acid as low as possible, as in Welman's method, in which alcohol is entirely or for the most part replaced by ether, ethyl acetate or anhydrous acetic acid. Waller (ANALYST, xx., 280) obtained the best results by adding strong hydrochloric acid, which acts not by combining with the water present, but by favouring the formation of iodine chloride and preventing its decomposition as in equation (2)

Of the substances taking part in the actual addition, free iodine is so slowly absorbed that it need not be taken into account in explaining the reaction. Like Ephraim (*loc. cit.*) the author formerly regarded iodine chloride as the chief agent in the addition, but has now come to the conclusion that the hypoiodous acid must be regarded as the principal substance. He argues that if iodine chloride were the most important, Waller's solution, which contains more iodine chloride, would act more rapidly than Hübl's solution, whereas experiments have shown the contrary to be the case. On the other hand, by shaking an alcoholic solution of iodine with finely divided mercuric oxide and filtering, the filtrate, according to Koene (*Pogg. Ann.*, lxvi., 302), contains mercuric iodide and hypoiodous acid, and with this solution correct iodine values were very rapidly obtained. A number of determinations of the iodine value of earthnut-oil, by means of Hübl's solution, to which various substances had been added, showed that when these substances favoured the formation of hypoiodous acid (*e.g.* iodine, mercuric chloride, and water), the halogen absorption took place more rapidly, whilst the converse was the case with substances which tended to repress its formation, such as hydrochloric acid and mercuric iodide.

In determinations of the iodine value there is invariably an increase in the "excess acid." The author considers that besides the addition of halogen, there is a further reaction with hydrochloric acid which, in the case of oleic acid, may be expressed thus:

$$CO_2H \cdot C_{17}H_{33}I \cdot OH + HCl = CO_2H \cdot C_{17}H_{33}ICl + H_2O.$$

But this only occurs without further change in strongly acid solutions like that of Waller. With solutions containing less acid there is a considerable increase in the "excess acid," which is to be accounted for by a splitting off of hydrochloric acid.

$$CO_2H \cdot C_{17}H_{33}ICl = CO_2H \cdot C_{17}H_{32}I + HCl.$$

The conclusions which the author has formed on this point are:

1. The separated acid is, *ceteris paribus*, proportional to the amount of absorption.

2. Under the same conditions different fats all yield about the same quantity of acid, proportional to the amount of fat taken. But with substances of a different constitution (allyl alcohol, cholesterol) different figures are obtained.

3. The greater the acidity of the solution, the smaller the quantity of acid separated.

4. The separation is most thorough in liquids which most readily dissolve hydrochloric acid. Thus it is greater in alcohol containing water than in chloroform, and in methyl alcohol than in ethyl alcohol.

In conclusion, the author summarizes the principal reactions which occur in the Hübl process in the following equations:

1. In the preparation:

$$HgCl_2 + 4I = HgI_2 + 2ICl$$
$$ICl + H_2O = HCl + HIO.$$

2. On keeping the solution:

$$2HIO + C_2H_6O = 2I + H_2O + C_2H_4O.$$

3. In the absorption (oleic acid):

$$CO_2H{\cdot}C_{17}H_{33} + HIO = CO_2H{\cdot}C_{17}H_{33}I{\cdot}OH.$$
$$CO_2H{\cdot}C_{17}H_{33}I{\cdot}OH + HCl = CO_2H{\cdot}C_{17}H_{33}ICl + H_2O.$$

And in a part of the fat:

$$CO_2H{\cdot}C_{17}H_{33}ICl = CO_2H{\cdot}C_{17}H_{32}I + HCl.$$

C. A. M.

A New Method of Determining the Iodine Absorption of Oils. J. J. A. Wijs. (*Berichte*, 1898, xxxi. [6], 750-752.)—In a former paper (ANALYST, preceding abstract) the author gave his reasons for concluding that in the Hübl process the substance chiefly concerned in the addition is hypoiodous acid, and he now suggests the more direct application of this acid as a means of improving the process. The fact, however, that it so readily decomposes ($5HIO = HIO_3 + 2H_2O + 4I$) prevents it being employed with any advantage at first hand. It was therefore found best to obtain the acid by the action of water on iodine chloride ($ICl + H_2O = HCl + HIO$), choosing a solvent which only contained so much of the former as would decompose nearly the whole of the latter, and which at the same time would not be oxidized by the hypoiodous acid. Good results were obtained with a solution of iodine chloride in 95 per cent. acetic acid. This was prepared by dissolving 13 grammes of iodine in a litre of the acetic acid, determining the "halogen content" of the solution and passing in a current of chlorine (free from hydrochloric acid) until the "halogen content" was doubled. With a little practice this point was readily hit by the change in colour. The solution thus prepared was more stable than that of Waller (ANALYST, xx., 280), and was employed precisely in the same way as Hübl's solution, with the exception that the length of time required for the absorption was very greatly reduced. In the case of oils with low iodine values, it was complete in three or four minutes, and with those with higher values not more than ten minutes was necessary, if too much oil had not been taken.

The following table gives the results obtained with this solution, compared with those of the ordinary Hübl process:

Oil.	Excess of Iodine. Per cent.	Time of Absorption.	I. Value.
Linseed	68	4 hours	180·91 (Hübl)
	57	5 min.	181·58
	57	7 ,,	182·25
	57	10 ,,	182·17
Liver-oil	61	4 hours	160·64 (Hübl)
	47	5 min.	164·79
	52	7 ,,	165·74
	65	8 ,,	166·51
	62	9 ,,	166·23
Maize ...	68	4 hours	124·87 (Hübl)
	65	3 min.	127·55
	64	6 ,,	128·56
	64	7 ,,	128·38
Poppy ...	69	4 hours	119·36 (Hübl)
	69	3 min.	119·66
	57	7 ,,	119·67
Sun-flower	70	4 hours	117·81 (Hübl)
	69	3 min.	118·92
	63	7 ,,	119·01
Sesame	69	4 hours	110·35 (Hübl)
	68	5 min.	111·87
	57	7 ,,	111·75
Cotton	68	4 hours	108·76 (Hübl)
	59	3 min.	110·07
	59	7 ,,	109·83
Rape	68	4 hours	102·96 (Hübl)
	65	3 min.	103·08
	61	7 ,,	103·33
Earth-nut	74	4 hours	87·26 (Hübl)
	70	2 min.	86·89
	70	3 ,,	87·13
	70	7 ,,	87·25
Olive	70	4 hours	83·27 (Hübl)
	70	3 min.	84·39
	70	7 ,,	84·45

In almost every case the values given by the new solution were higher than the ordinary Hübl values, but the author considered them the more correct by reason of the results obtained with purified allyl alcohol. This has a theoretical iodine value of 435. By Hübl's process Lewkowitsch obtained values varying from 349 to 376. Using an excess of 75 per cent. of iodine, the author found it to have an iodine value of 425 by Hübl's process, whilst with iodine chloride in acetic acid (the excess of iodine being the same) his results were: after five minutes 434·1 and after ten minutes 436·8. C. A. M.

Candle-Nut Oil. G. de Negri. (*Oesterr. Chem. Zeit.*,* 1898, i., 202.)—The following particulars respecting this substance were derived from the examination of two small samples extracted from seeds by the author himself: one with ether, the other with petroleum spirit. The yield was 62·25 per cent.; the product a bright, transparent, pale-yellow oil, which soon became rancid, and dried only a little more readily than tung-oil. It was soluble in ether, petroleum spirit, and chloroform; insoluble in cold absolute alcohol, with which it yielded a turbid mixture, but slightly soluble in hot alcohol. It did not dissolve in (? cold) glacial acetic acid; at the boiling-point it was soluble in equal volumes. It remained liquid at a temperature

* This is the same journal that was formerly known as the Zeitschrift für Nahrungsmittel-Untersuchung, Hygiene, und Warenkunde; and the corresponding reference is 1898, xii. [14], 202.

of – 18° C. It was readily saponified by alcoholic alkali, the soap being easily soluble in water. Some of the constants are given herewith:

	Extracted with Ether.	Extracted with Petroleum Spirit.
Specific gravity at 15°	0·926	0·920
Saponification number	187·36	184·0
Iodine number	139·34	136·29
Melting-point of fatty acids	20-21°	—
Solidification point of fatty acids...	13°	—
Iodine number of fatty acids	144·13	142·71
Refractometer number at 15° (Zeiss-Wollny) ...	—	76-75·5

With Brullé's test it gave no colour in the cold; on warming, a red which was permanent for twenty-four hours, while the oil became thick. Baudoin's test produced no colour. With Becchi's test the oil extracted by light petroleum yielded an intense brown; that recovered with ether only a faint brown. When exposed to the direct rays of the sun in sealed tubes for twenty-five days, both specimens of candle-nut oil, and also extracted tung-oil, remained perfectly liquid; whereas expressed tung-oil polymerized and formed some solid matter. Candle-nut oil recovered by carbon disulphide remained fluid when treated as above; tung-oil obtained by the same solvent solidified.

The material described by Laach (*Chem. Zeit.*, 1890, 871) was probably a mixture of fatty acids from different kinds of *aleurites*, for his figures are very unlike those observed by De Negri. F. H. L.

Improvements in Organic Combustion. F. Swarts. (*Chem. Zeit.*, 1898, xxii., 474.)—This process was originally devised for the ultimate analysis of compounds containing fluorine, but it is equally well adapted for all classes of organic bodies. In place of the ordinary glass tube, a seamless tube of copper is employed, about 1·5 metre long, 12 to 18 mm. in diameter inside, with walls 3 or 4 mm. thick. It is first cleaned by being ignited in a furnace while a current of air is passing through it; by which means also a coating of oxide is formed that materially assists in the combustion. In order to protect the corks at either end from being injured by the excessive heat due to the high conductivity of the metal, short jackets (about 12 to 15 cm. × 3 to 4 cm. in size) with corks and leading tubes are applied close to both extremities, and water is kept circulating through them. The combustion tube is charged much in the usual fashion; but instead of using copper oxide alone the author prefers a mixture of three parts of CuO with one of granulated PbO, the latter prepared by heating pure red lead to incipient fusion in a copper basin with constant stirring. The tube is laid in a Glaser furnace; the substance to be burnt in a boat. At the point where the boat stands, the bent iron bottom-plate of the furnace is removed, and its place is taken by a somewhat more deeply curved casting having a hole and leading tube at its lowest point. Immediately over the tube at the same spot is a short piece of copper pipe closed at each end, perforated underneath with many fine holes, and connected to a water-supply. By the aid of

a tap a stream of water (which afterwards runs away through the hole just mentioned) may be caused to play on the portion of the main combustion tube that surrounds the boat; and, by regulating or stopping the supply, the substance can be volatilized in the current of purified air at any desired speed. In this manner much time is saved, for the copper-lead oxide can be brought to a red heat while the weighings are in progress, and the organic body maintained cold when in position until the roll of oxidized copper gauze at the other end of the tube is also hot. When the combustion seems to be nearly finished, the rubber tubes for the delivery and exit of the water are disconnected, and the part of the copper tube round the boat is brought to a red heat for five or ten minutes in order to burn up the residual carbon. Some of the water from the oxidation of the material is condensed at the end of the tube by reason of the short condenser there fixed. This may be driven over into the absorbing vessel by stopping the current of water, emptying the jacket if necessary till the deposited moisture boils again; but the amount retained within the tube may be reduced to a minimum by so regulating the current that the extreme ends of the copper are kept slightly warm all through the operation.

Not only can the copper tube be raised to the working temperature almost instantaneously, but the speed of the combustion itself is under ready control. Compared with these advantages, the lack of transparency is not a serious obstacle, for the progress of the operation can be gathered by watching the potash bulbs. The coating of copper oxide in the tube prevents the deposition of very resistant carbon; and the tube itself lasts for about sixty analyses before being worn out.

F. H. L.

Kjeldahl's Method and its Modifications. A. Atterberg. (*Chem. Zeit.*, 1898, xxii., 505.)—Reviewing seven different modifications of the original Kjeldahl process in a recent work on chemistry, the author stated that Kellner's seemed to be the best and quickest. The matter has now been further investigated, using as a test-substance a bog-earth containing 15 per cent. of moisture and 7 per cent. of ash, and conducting the oxidation as fast as possible in a 250 c.c. flask. Gunning's method occupies ninety-five minutes, and much frothing occurs; if a drop of mercury is added, the time is roughly halved; if the potassium sulphate is not introduced till the material is dissolved (the proportion between the H_2SO_4 and the K_2SO_4 being kept as specified by Gunning), the process only takes about twenty-six minutes, and no foaming is noticed. Kellner's method requires forty-five minutes, which may be reduced to twenty-seven by the employment of potassium sulphate. The latter substance therefore is just as efficient as Kellner's phosphoric anhydride—both raise the boiling-point of the sulphuric acid; and orthophosphoric acid itself might be used instead of the anhydride were it not for the separation of mercury phosphate, which interferes with the boiling of the liquid. Experiments have been tried with metals or oxides other than mercury, in order, if possible, to avoid the necessity for adding sulphide when distilling off the ammonia. Only molybdic acid (*cf.* ANALYST, xxi., 267) has been found to work without frothing; but as the oxidation is made a trifle slower, the final solution is blue instead of colorless, and commercial specimens of the reagent often contain much nitrogen, it can hardly replace mercury.

The author quotes some figures obtained with sulphate of quinine which go to prove that the best oxidizing solution is composed of 20 c.c. of strong sulphuric acid, 15 to 18 grammes of potassium sulphate, and a little mercury. When frothing is expected, the sulphate should not be introduced till the nitrogenous body is dissolved, otherwise it may be added at the outset. Decolorization takes place (in a 250 c.c. flask) in about 30 minutes; but the whole should be boiled 15 minutes longer.

F. H. L.

Detection of Sulphur and of Nitrogen in Organic Bodies by means of Phloroglucinol and Vanillin. P. N. Raikow. (*Chem. Zeit.*, 1898, xxii., 377.)—The red colour which is produced in a dried alcoholic solution of phloroglucinol and vanillin by the action of the halogens has already been described (ANALYST, xxiii, 99). A similar reaction is determined by sulphur, phosphorus, and arsenic; and in the case of sulphur it is so general that the test is available for the examination of organic substances of whatever kind. It shows the presence of thiophen in benzene, and it distinguishes between the proportion of the impurity in German, Russian, and Pennsylvanian petroleum. Sulphuretted hydrogen does not yield the red colour; it is derived from sulphurous oxide, and the test is so delicate that if methyl thiocyanate is diluted with alcohol, and a drop equivalent to 0·001 milligramme of sulphur is burnt, the coloration is still visible.

As Becchi's test for cotton-oil depends on the sulphur it contains, experiments have been made (and are still unfinished) on various oils with the present reagent. Sesamé, castor, walnut, olive, arachis, white cod-liver, and huile de Veillette, give no colour. Cotton, colza, linseed, rape, and seal, yield a more or less intense red. The colour produced by linseed-oil is much fainter than that of colza or cotton-oil, and the degree of volatility of the compound which contains the sulphur varies largely in different oils, that in cotton-oil having such a high volatilizing point that the strength of the reaction is not diminished when the oil is treated with steam for twelve hours. All of these oils contain but little sulphur, and as many burn with a very smoky flame, the test is rather difficult to carry out. The sample is best burnt in a glass tube bent at a right angle close to the end; in the bend is placed some long fibre asbestos to act as a wick, and the size of the flame is regulated by altering the position of the tube. If the flame be kept quite small and held very near the lid of the porcelain crucible which bears the dried film of reagent, the colour soon appears; but if the soot is still troublesome the oil may be diluted with ether or alcohol.

All kinds of albumin, fibrin and casein give a fine red colour, as do also all varieties of paper, even filter-paper.

The behaviour of nitrogenous organic matter with the mixture of phloroglucinol and vanillin depends on whether the compounds evolve their nitrogen as acid, ammonia, or in the free state, when they are broken up. If a few drops of the reagent are mixed with one drop of nitric acid, and the whole set light to, an intense red colour is produced; under similar conditions ammonia gives a bright yellow. No colour at all is formed when any nitrogenous substance is held in a loop of platinum-wire in a powerful Bunsen flame under the porcelain lid, because the nitrogen is set

free. No colour is produced if nitro- or nitroso-compounds or nitriles are allowed to burn by themselves; but amines and similar bodies give a yellow. Aromatic compounds in which the nitrogen is part of the closed chain as a rule give no yellow, *e.g.*, quinoline, piperidine, etc., but antipyrin and isatin yield faint yellows. The test will show 1 per cent. of aniline in quinoline.

When nitrogenous bodies which give the yellow reaction also contain sulphur or a halogen, the red colour is produced as well, *e.g.*, thiourea, chloraniline, methyl violet, etc., also wool, hair, feathers, casein. But sometimes a faint red is to be found even in the absence of sulphur and chlorine, as part of the nitrogen has burnt to acid; in this case the sample should be decomposed in the Bunsen flame, when no red will be observed.

Raikow prefers to dissolve the phloroglucinol and vanillin in ether rather than in alcohol, as the solution is more permanent, and also to use it much weaker than recommended by Günzburg. F. H. L.

Remarks on the Estimation of Tannin, and on the Titration of Tannin and Gallic Acids. F. Jean. (*Ann. Chim. Analyt.*, vol. iii. [5], pp. 145-149.)—The author confirms the observation made by Raŭ (*Jour. Amer. Chem. Soc.*, vol. ix., No. 4), that up to 50 per cent. of gallic acid is fixed by hide powder and other gelatin-yielding tissues, and also demonstrates that, in order to more closely approximate laboratory results to those obtained in tannery practice, the Hammer method requires modification to render it suitable for the estimation of tannin and substances which can be fixed by the hide. With this object he proposed to replace the treatment with hide powder by forty-eight hours' maceration with finely-shredded rabbit-skin—the anatomical structure of which has not been destroyed—since he finds that by this means the fixation of gallic acid is reduced from 42 per cent. to 27 per cent.

Similarly, the Hammer method is unsuitable for determining the tannin and gallic acid in astringent bodies, such as sumach. In this case, albumin proves the best means of separating these two substances, a sumach that yielded 2·396 per cent. of gallic acid when treated with hide powder furnishing 13·4 per cent. of the acid by the albumin method. The loss of gallic acid observed when working with a mixture of known composition is reduced by this latter method to 2·2 per cent.

C. S.

Method of Preparing Pure Sugar. H. Pellet. (*Bull. Assoc. Chim. de Sucr. et Distill.*, vol. xv., p. 813; through *Rev. Chim. Analyt. Appl.*, vol. vi. [9], p. 143.)—Five hundred grammes of refined loaf-sugar are dissolved in 250 grammes of distilled water and filtered if necessary. To the clear solution 560 to 570 c.c. of absolute alcohol are added by degrees, the liquid being agitated to prevent crystallization, and afterwards covered up and set aside for several days to allow the excess of sugar to crystallize out. The supernatant liquid is then poured off, and the crystals—which are loosened from the walls of the vessel by immersing the latter in warm water—are collected in a tapped funnel, and washed with alcohol increasing in strength from 70 per cent. to 100 per cent. Finally, they are dried over sulphuric acid, and a

10-gramme sample is incinerated to test the freedom from ash. If found impure, the crystallization process is repeated. C. S.

INORGANIC ANALYSIS.

The Separation of Mercury and Bismuth. L. Vanino and F. Treubert. (*Ber.*, 1898, xxxi., 129, 130.)—This is based on the fact that on adding to a dilute hydrochloric acid solution of mercuric chloride and bismuth oxychloride a mixture of hypophosphorous acid and hydrogen peroxide (1 drop of the former to 1 c.c. of the latter), the mercury is separated quantitatively as calomel, while the bismuth remains in solution, and can be precipitated by the further addition of hypophosphorous acid. The details of the method are as follow:

The solution of the mixed salts is treated with the reagent as above, and, after standing for about an hour, the precipitated calomel is filtered off, rapidly washed free from bismuth, first with dilute hydrochloric acid, and subsequently with cold water, and dried at 105° C.

The filtrate is made slightly alkaline with sodium hydroxide, warmed over a naked flame, and hypophosphorous acid added until no more black precipitate is produced, and no turbidity occurs in the supernatant liquid on the addition of water. The precipitate is pressed into a compact mass with a glass rod to prevent oxidation during the drying, collected on a Gooch's filter, washed successively with water and alcohol, and dried at 105° C.

A table giving the results of the method applied to a test mixture shows that the figures obtained in this way are in close agreement with those required by theory.

C. A. M.

Qualitative Separation of the Metals of the Iron, Chromium, etc., Group, and of the Rare Earths. G. Posetto. (*Giorn. di Farm. di Trieste*, 1898, iii., 70; through *Chem. Zeit. Rep.*, 1898, 135.)—The original solution is treated with ammonia and ammonium sulphide, and separated into a filtrate (A) and a precipitate (B) containing the hydrates of Fe, Cr, Al, Ce, La, Th, Zr, Y, U, Be, Di, the sulphides of Mn, In, Ni, Co, and the phosphates, etc., of Ba, Sr, Ca, and Mg. This is warmed with 1 : 5 HCl, which dissolves everything except the Ni and Co. The solution (C) is mixed with nitric acid, boiled for a time, then cooled, and made just alkaline with strong KHO. Only the Be is left unprecipitated, and it is thrown down on diluting the liquid with three or four times its volume of water and then boiling. The precipitate (D) is boiled in KHO to remove Al and Zn, and the insoluble matter (E) is washed and digested in ammonium carbonate solution with occasional agitation for twelve hours. The filtrate (F) contains the carbonates of Ce, Th, Zr, La, U, Di, and Y; the residue (G) Mn, Fe, Cr, and Ba, Sr, Ca, Mg as phosphates, borates, etc. After the Mn, Fe, and Cr have been specially tested for in small quantities of (G), the main portion is boiled for ten minutes with sodium carbonate solution, which decomposes the bulk of the phosphates, etc., of the alkaline earths into carbonates, giving a solution (H) and a precipitate (L). The latter is dissolved in HCl, precipitated with ammonia and

sulphide, the insoluble portion is thrown aside, and the filtrate, mixed with the first solution (A), is examined for alkaline earths and alkalies as usual.

(F) is boiled till the carbonates are deposited and the liquid yields no residue on evaporation. The precipitate (N) is divided into different parts: (1) Is mixed with hydrogen peroxide—in presence of Ce the white powder changes to yellow—or it is boiled with excess of nitric acid and lead peroxide, when the liquid becomes yellow. (2) Is treated in a porcelain basin with a few crystals of iodine—La causes the mass to turn blue. (3) Is dissolved in water containing a trace of HCl and subdivided into three. Turmeric paper is dipped into (3*a*) and dried at 100° C.—a red colour like that produced by boric acid indicates Zr. (3*b*) Is tested with potassium ferrocyanide—U gives a red colour or precipitate. (3*c*) Is treated with sodium carbonate —Di forms a white precipitate insoluble in excess. A fourth portion of (N) is dissolved in weak HCl, neutralized with KHO, a few crystals of normal potassium sulphate added, and the whole boiled. Th, Zr, and Ce are thrown down as double sulphates (O). The filtrate is again boiled several times to ensure the removal of all insoluble matter; but Y remains in solution, and may finally be recovered on addition of ammonia. The precipitate (O) is boiled in HCl and water—the double sulphates of Th and Ce dissolve, that of Zr remains unattacked. The solution is treated with oxalic acid to throw down the earths, and the precipitate is boiled in strong ammonium oxalate till it dissolves. After cooling, the liquid is diluted with three times its volume of water, which deposits the oxalate of Ce. The Th is recovered from the filtrate by treatment with ammonia. F. H. L.

Precipitation of Platinum from its Solutions. A. Atterberg. (*Chem. Zeit.*, 1898, xxii., 538.)—Of the numerous substances which may be employed to throw down metallic platinum from its solutions, thiacetic acid, mercury, and magnesium are the most useful. The first yields a particularly voluminous precipitate, and it is therefore to be recommended when only traces of the metal have to be dealt with. When it is desired to estimate the sodium in the alcoholic filtrate from a potash determination mercury is the best reagent. The platinum solution is concentrated to a small volume on the water-bath in a deep porcelain basin, an excess of mercury is added, and the whole is well stirred till the reaction is complete. The by-product is mercuric (not mercurous) chloride, and can easily be removed by heat. The reduction only takes place in the warm; the platinum is deposited as a compact powder, but the operation must never be conducted in a platinum vessel, as the metal sticks firmly to the walls. If the platinum which is in solution has to be weighed, the best precipitant is magnesium ribbon. The liquid may be neutral, but it is better acidified with a little hydrochloric acid. The deposition proceeds slowly in the cold, more quickly on warming or on agitation; but a trace of metal remains in solution for some time, giving it a grayish colour, so that the liquid should not be filtered until it is water-white. The platinum is heated with HCl, washed very thoroughly, and gently ignited.

For working up alcoholic platinum residues magnesium ribbon is also most

convenient. In twenty-four hours all the metal is thrown down, and can be decanted off, purified with acid, and washed. F. H. L.

Separation of Nickel and Cobalt with Potassium Nitrite. Schlagdenhauffen and Fageot. (*Bull. commerc.*, 1898, xxvi., 225; through *Chem. Zeit. Rep.*, 1898, 170.)—When describing this process, the books usually require that the solution of the metals shall be treated with an equal volume of nitrite and an excess of acetic acid. The present authors find that this is not sufficiently explicit to ensure uniform results. The strongly concentrated liquid containing the metallic chlorides must be mixed with a cold-saturated solution of potassium nitrite, diluted with at least five times its volume of water, and 15 to 30 c.c. of acetic acid added for every 1 gramme of chloride present. The nickel remains in solution and the cobalt is completely precipitated, so that the method serves for their quantitative separation, or for the detection of traces of either one in the other. F. H. L.

The Colorimetric Estimation of Small Amounts of Chromium. W. F. Hillebrand. (*Jour. Amer. Chem. Soc.*, 1898, xx., 454-460.)—The chromium is brought into solution as monochromate, the liquid rendered alkaline with sodium carbonate, and compared with a standard solution of chromate, also made alkaline with sodium carbonate. Two standards are prepared, containing 0·25525 and 0·5105 gramme of potassium chromate per litre respectively, a little sodium carbonate being added to each. One c.c. of the first solution corresponds to one-tenth of a milligramme of chromium oxide.

In testing the applicability of the method to the determination of chromium in rocks and ores, varying amounts of the standard solution were evaporated in a crucible with 5 grammes of an iron ore containing phosphorus and vanadium, and the residue fused with 20 grammes of sodium carbonate and 3 grammes of sodium nitrate. The aqueous extract was nearly neutralized with nitric acid after the reduction of the manganese with alcohol, and was evaporated so as to approximately separate the silica and alumina. In order to recover any chromium carried down with these, the precipitate was ignited, the silica removed by treatment with sulphuric and hydrofluoric acids, the residue fused with sodium carbonate, the alumina separated as before, and the filtrate added to the first filtrate. Mercurous nitrate was then added, and the resulting precipitate, containing phosphate, chromate, vanadate, and carbonate of mercury, was slightly washed and ignited. The residue was fused with sodium carbonate, extracted with water, filtered, the solution made up to 50 or 100 c.c. and the colour compared with the standard, as in a Nessler determination of ammonia. A similar determination was made with a silicate rock.

	Chromic Oxide added. Milligrammes.	Chromic Oxide found. Milligrammes.
1. Five grammes iron ore ...	7·03	7·18; 7·20; 7·25; 7·21
2. ,, ,, ...	2·99	3·08; 3·13
3. Two grammes silicate ...	1·6	1·53; 1·57; 1·59

When the percentage of chromic oxide in an ore exceeds two-tenths per cent., and a vanadium determination is not required, the colour of the original extract of the carbonate fusion may be matched after reduction and removal of the manganese, but when much less than that quantity is present the precipitation with mercurous nitrate is necessary to obtain a sufficiently colored filtrate in a small bulk.

C. A. M.

The Volumetric Determination of Vanadium in the Presence of Small Amounts of Chromium. W. F. Hillebrand. (*Jour. Amer. Chem. Soc.*, 1898, xx., 461-465.)—When chromium and vanadium occur together, and the former has been estimated colorimetrically (see preceding abstract), the vanadium can in many cases be determined without separation from chromium by reduction with sulphurous acid and titration with permanganate in sulphuric acid solution. The one limitation to the method is that too much chromium must not be present, since it interferes with the end-reaction; but satisfactory determinations of as little as 1 or 2 milligrammes of vanadium pentoxide can be made in the presence of 30 milligrammes of chromic oxide. The method is not applicable in the presence of molybdenum, and arsenic, if present, must be removed by precipitation with sulphuretted hydrogen.

The rock or ore (free from arsenic) is fused with sodium carbonate and nitrate, the melt extracted with water, the manganese reduced, the silica and alumina removed, and the chromium determined colorimetrically, as described in the preceding abstract.

After the colorimetric determination, sulphuric acid is added, the chromium and vanadium reduced by means of sulphur dioxide, and the excess of the latter expelled by boiling the liquid in a rapid current of carbon dioxide. When cooled to about 70° or 80° C., the solution is titrated with a very dilute standard solution of potassium permanganate, of which 1 c.c. corresponds to about 0·001 gramme of vanadium pentoxide as calculated from its equivalent in iron.

The subjoined table gives the results of test analyses of an iron ore and silicate rock to which known amounts of chromium and vanadium had been added:

	Chromic Oxide added. Milligrammes	Vanadium Pentoxide added. Milligrammes.	Vanadium Pentoxide found. Milligrammes.
Iron ore, 5 grammes ...	7	6·76	6·81; 6·48; 6·43; 6·37
,, ,, ...	8	3	3·08; 3·13; 3·03
Silicate, 2 grammes ...	1·6	1·87	1·86; 1·97; 2·07; 1·86

C. A. M.

The Iodometric Estimation of Tellurium. J. F. Norris and H. Fay. (*Amer. Chem. Jour.*, 1898, xx., 278-283.)—Attempts were made to employ the volumetric method found satisfactory in the case of selenium (ANALYST, xxii., p. 82) in the estimation of tellurium. One molecule of tellurous acid reacts with four of sodium thiosulphate in the presence of dilute acid, but the end-point is not sharp, since the addition of the thiosulphate produces a yellow solution, and the reduced tellurium compound is slowly oxidized by iodine.

A more successful method was found in the oxidation of tellurous acid to

telluric acid by means of potassium permanganate, and determination of the excess of the latter by adding potassium iodide and sulphuric acid, and titrating the liberated iodine with thiosulphate.

The reactions involved in the process are:

$$(1)\ 2KMnO_4 + 3TeO_2 = K_2O + 2MnO_2 + 3TeO_3,$$

in which the manganese is not precipitated, but remains in solution probably as a manganite or in combination with the telluric acid.

On adding to such a solution potassium iodide and sulphuric acid, the following change occurs:

$$(2)\ 2MnO_2 + 4KI + 4H_2SO_4 = 2MnSO_4 + 2K_2SO_4 + 4H_2O + 4I.$$

Any excess of permanganate reacts thus:

$$(3)\ 2KMnO_4 + 10KI + 8H_2SO_4 = 2MnSO_4 + 6K_2SO_4 + 8H_2O + 10I.$$

Combining equations (1) and (2), the oxidation of tellurium dioxide may be thus expressed:

$$(4)\ 2KMnO_4 + 3TeO_2 + 4KI + 5H_2SO_4 = 3TeO_3 + 3K_2SO_4 + 2MnSO_4 + 5H_2O + 4I.$$

The precautions to be observed are that the oxidation must take place in the presence of a large excess of alkali, and that the solution must be dilute and cold in order to prevent the reduction of the telluric acid by the hydriodic acid.

A sample of pure tellurium dioxide analyzed in this way gave eight results varying from 79·80 to 80·15 per cent. of tellurium, as against 79·94 per cent., the theoretical amount. The accuracy of the method in the presence of halogen acids was proved by the analysis of the double bromide of tellurium and potassium with which four consecutive results (18·58 to 18·64 per cent.) were obtained, the theoretical percentage of tellurium being 18·61.

Experiments proved that this method is also applicable to the determination of selenium, but it offers no advantages over the simpler method proposed before.

C. A. M.

Estimation of Various Sulphides by Titration with Permanganate. J. **Hanus.** (*Zeits. anorg. Chem.*, 1898, xvii., 111.)—The sulphides of antimony, lead, and bismuth are oxidized by treatment with ferric sulphate, and the reduced ferrous salt can be titrated with permanganate in the usual way. Tin and cadmium behave similarly, as mentioned by Mohr; but the reaction with copper is not complete, and the sulphides of mercury and arsenic cannot be determined in this manner. About 0·2 or 0·3 gramme of precipitated antimony trisulphide is washed into a beaker and boiled for 15 minutes with excess of powdered ferric sulphate; after cooling, sufficient strong sulphuric acid (15 c.c.) is added to dissolve the precipitate, and when this is effected the clear liquid is made up to 200 c.c., filtered through a dry filter, and 100 c.c. taken for titration with the permanganate. Free sulphur does not affect the process; but the precipitate must be entirely dissolved in the sulphuric acid, and it must not be exposed to air, or some inert red oxysulphide may be produced. The reaction is as follows:

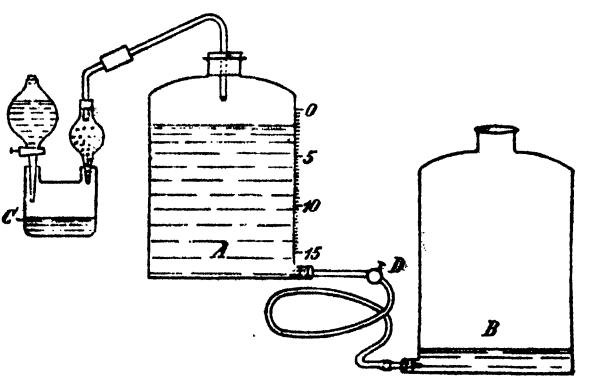
$$Sb_2S_3 + 5Fe_2(SO_4)_3 + 8H_2O = 2H_3SbO_4 + 10FeSO_4 + 5H_2SO_4 + S_3.$$

With lead the process works satisfactorily, provided not more than 0·3 gramme of PbS is taken. Two molecules of ferrous sulphate result from the oxidation of one of lead sulphide. In the case of bismuth sulphide, which is attacked with great ease, the proportion is $Bi_2S_3 = 6FeSO_4$. F. H. L.

Solubility of the Bicarbonates of Calcium and Magnesium. F. P. Treadwell and M. Reuter. (*Zeits. anorg. Chem.*, 1898, xvii., 170.)—This article contains an account of various experiments on the bicarbonates of the alkaline earths which were carried out to determine whether these compounds actually exist as such, and also to estimate their solubility in water in the presence or absence of free carbon dioxide. The results given briefly are: Calcium bicarbonate does exist as such in aqueous solution, its solubility at 15° C. and average atmospheric pressure being 0·385 gramme per litre. The solubility of calcium bicarbonate is not appreciably affected by dilute (decinormal) solutions of common salt. Magnesium bicarbonate does not exist in solution except in the presence of free carbon dioxide; in the absence of the gas it breaks up into a mixture of normal carbonate and bicarbonate, until the liquid, at the above temperature and pressure, contains 0·7156 gramme of the former and 1·954 gramme of the latter per litre. F. H. L.

APPARATUS.

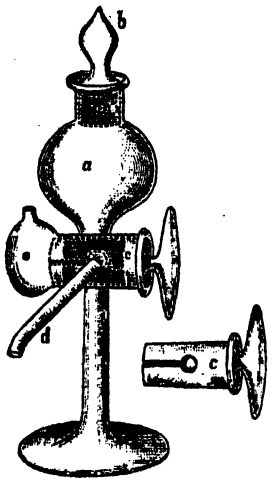
An Apparatus for the Analysis of Calcium Carbide. H. Bamberger. (*Zeit. angew. Chem.*, 1898, 243.)—This is recommended for the simple and accurate valuation of calcium carbide by a method combining the author's gravimetric process (ANALYST, this vol., p. 167) with the gas-volumetric process of Fuchs and Schiff (ANALYST, this vol., p. 25).



The flask A, which serves as a gasometer, contains 20 litres, and is graduated into decilitres. It is connected with a similar flask, B, and is nearly filled with a concentrated solution of sodium chloride, saturated with acetylene at the ordinary temperature. The gas is generated in the gravimetric flask, C, and drives the brine forward into B. When the reaction is over, the liquids in both flasks are brought to the same level, and the amount of acetylene read off and corrected for temperature and pressure (*cf.* ANALYST, this vol., p. 25).

The apparatus can be obtained from Max Kähler and Martini, Berlin, W.

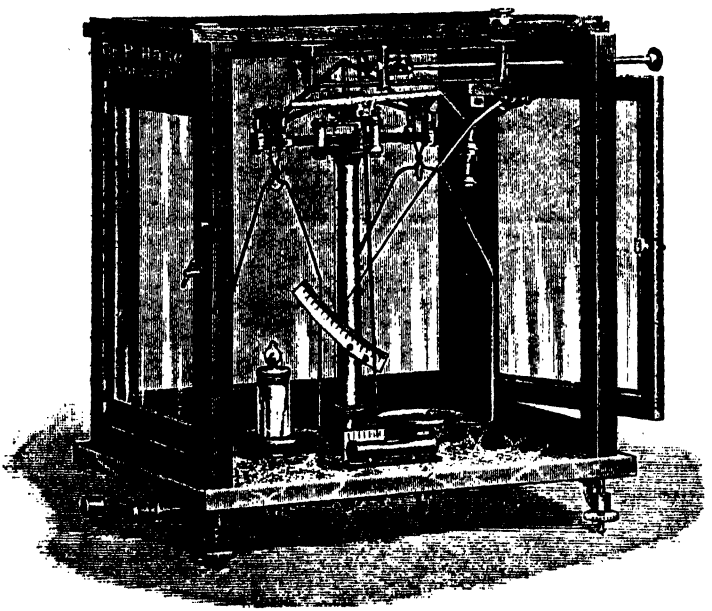
C. A. M.



Apparatus for the Delivery of Small Quantities of Mercury (for Nitrogen Determinations). R. Meyer. (*Chem. Zeit.*, 1898, xxii., 331).—As shown in the accompanying illustration, this apparatus serves for the storage of about 250 grammes of mercury in the bulb *a*, and also for the delivery of a definite amount of the reagent such as is required in a Kjeldahl nitrogen estimation. When the hemispherical depression in the stopcock *c* is in a vertical position it fills with mercury from the reservoir, and when it is rotated till opposite the exit tube *d* the mercury runs out, its place being taken by air entering through *e* and the groove in the cock. *e* also catches any drops that may escape out of the groove while the hole is being filled.

F. H. L.

An Improved Analytical Balance. (*Chem. Zeit.*, 1898, xxii., 540.)—This apparatus consists essentially of a short-beam analytical balance, which, in order to gain an approximate idea of the weight of any substance placed in the pan, can be temporarily put in connection with a spring-balance by means of the knob seen projecting from the top of the case in the accompanying illustration. When the spring-balance is in gear, the long lever shows the weight of the substance on the curve scale to the nearest deci- or centigramme; and, after it has been thrown out, the correct weights can usually be placed at once on the opposite pan, leaving only the rider of the balance proper to be manipulated. The convenience of this arrangement is apparent, and it will be specially appreciated in weighing out exact quantities of things, for during the whole process the arresting-gear of the true balance need not be used at all (see also ANALYST, xxi., 248).



F. H. L.

THE ANALYST.

OCTOBER, 1898.

PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS.

THE PROPORTION OF OXYGEN PRESENT IN LINSEED-OIL, BOTH BEFORE AND AFTER OXIDATION.

By Rowland Williams.

(Read at the Meeting, June 1, 1898.)

The elementary composition of raw linseed-oil is stated by W. T. Brannt ("Animal and Vegetable Fats and Oils," p. 273) to be as follows:

	C.	H.	O.
Cold-drawn	78·11	10·96	10·93
Hot-pressed	75·27	10·88	13·85

In Allen's "Commercial Organic Analysis," vol. ii., p. 118, I find the following figures for linseed and poppy oils respectively, the results of the linseed-oil being those of three observers:

Observer.	Kind of Oil.	C.	H.	O.
Sacc	Linseed	78·11	10·98	10·93
König	,,	77·40	11·10	11·50
Clöez	,,	77·57	11·33	11·10
,, ...	Poppy	77·50	11·40	11·10

After eighteen months' exposure the two last samples gave:

Clöez	Linseed	72·27	10·57	24·16
,, ...	Poppy	71·38	10·64	25·08

These figures indicate comparatively little difference between linseed and poppy oils, either in the raw state or after "drying."

About a year ago I was engaged upon a research, in the course of which it was necessary to submit to ultimate analysis several samples of linseed-oil, some raw and others after boiling and oxidizing in various ways. The results are given in the table below:

	Raw.		Boiled.				Solid.			
	1.	2.	3.	4.	5.	6.	7.	8.	9.	10.
C. ...	75·03	75·40	74·66	74·32	69·74	69·52	64·74	65·40	68·64	64·38
H. ...	10·78	10·64	10·38	10·04	9·57	9·49	9·01	9·00	9·24	9·01
O. ...	14·19	13·96	14·96	15·64	20·69	20·99	26·25	25·60	22·12	26·61

The figures in the first two columns were obtained from raw linseed-oil, those in the third from moderately stout boiled oil. All the other figures refer to samples of solid oil, such as is used in the manufacture of linoleum. Sample 4 was made by one method, 5 by another, samples 6 to 9 by a third process, and 10 by a fourth.

As will be observed, sample 4 contains very much less oxygen than any of the other samples of solid oil. It is interesting to notice that the samples marked respectively 8 and 10 gave fairly similar results, although the oils were made by entirely distinct methods.

NOTE ON CERTAIN RESINS.

By Rowland Williams.

(*Read at the Meeting, June* 1, 1898.)

Some years ago I made an investigation of a number of resins and gums, the results of which are recorded in the *Chemical News*, vol. lviii., pp. 224, 225. I have recently examined, on somewhat similar lines, samples of kauri, copal, and dammar, representing large stocks of these particular resins. The chief point of interest about the present samples lies in the fact that the kauri and one of the copals were examined not only in their original condition, but also after being subjected to the melting or "running" process, which is applied to certain resins previous to converting them into varnish.

	Kauri.		Copal.			Dammar.
			Fine Quality.		Finest.*	
	Ordinary Condition.	After "Running."	Ordinary Condition.	After "Running."	Ordinary Condition.	Ordinary Condition.
	Per cent.	Per cent.	Per cent.	Per cent.	Per cent.	Per cent.
KOH absorption, hot	6·42	5·16	18·87	13·78	12·87	2·85
,, ,, cold	6·19	4·76	14·36	10·99	11·56	2·63
Iodine absorption ...	130·62	126·52	124·32	129·60	95·40	99·40
Loss or gain on heating at 100° C. for fifteen hours ...	11·81 loss	0·80 gain	0·82 loss	2·26 gain	0·74 loss	0·84 loss
Mineral matters ...	0·15	0·14	0·14	0·16	0·03	0·05

As would be anticipated, the resins in their original state lost weight on heating at 100° C.—the kauri to a considerable extent, the others only slightly. It is interesting to observe, however, that the two resins which had been fused, when heated under the same conditions, gave a decided increase in weight, especially the copal, pointing to a certain amount of oxidation having taken place. It may be well to mention that the hot KOH determinations were made according to Koettstorfer's method, but, as some resins are rather difficult to attack completely, a very large

* This is an exceptionally fine quality of copal, used only in the manufacture of the palest and highest class of varnishes.

excess of alcoholic KOH was added, and the boiling continued for a much longer time than usual.

The cold KOH determinations were made by digesting the finely powdered resins with a mixture of ether and alcohol, and titrating with $\frac{N}{2}$KOH, using phenolphthalein as indicator. The percentages of iodine absorbed were estimated as in the case of oils and fats, a large excess of Hübl's reagent being allowed to act all night (sixteen hours).

EXPERIMENTS ON THE EFFECT ON BUTTER OF FEEDING COTTON-SEED AND SESAME OILCAKE.

(*Reprinted from the Annual Report on the Distribution of Grants for Agricultural Education and Research in the year* 1897-98.)

EXPERIMENTS on behalf of the Board of Agriculture were undertaken during the present year by the South-Eastern Agricultural College, at Wye, for the purpose of determining whether, and to what extent, the substances giving the cottonseed and sesame oil reactions might be found in the butter made from the milk of cows fed on cottonseed and sesame oilcakes.

The following report on the results of the experiments has been drawn up by Dr. T. E. Thorpe, F.R.S., the principal chemist of the Government Laboratories.

The experiments were carried out as follows:

Twelve cows (particulars of age, breed, etc., are given in Appendix A) were divided into three sets of four. Commencing on the 16th of February last, all the cows were fed with the standard ration described in Appendix B, together with 4 lbs. of linseed oilcake each per day. This feeding was continued till the 19th of March. Milk was taken at intervals of a day or two from each cow, and made into butter under the supervision of the Principal of the Wye College. Samples of the butter were forwarded to the Government laboratory, where they were examined for the cotton-seed and sesame oil reactions. The results of the tests were uniformly negative; and it having been established that none of the cows gave any indications of cotton-seed or sesame reactions in the absence of the cake, the feeding with cotton and sesame cake was commenced. On the 19th of March one half the daily allowance of linseed oilcake was replaced by a similar amount of cottonseed oilcake in the case of the first set of cows (Nos. 1 to 4), and by sesame cake in the case of the second set (Nos. 7 to 10), the third set (Nos. 5 and 6, 11 and 12) being continued on the linseed cake as before for purposes of comparison.

On the 20th of March samples were obtained from each cow as before. On the examination of the butter, the samples from the first set of cows (Nos. 1 to 4) gave a distinct reaction for cottonseed-oil by two tests. No such reaction was given by any of the other samples, nor was any reaction for sesame-oil given by the samples from any of the cows. Samples taken at intervals during this period gave similar results. On the 28th of March the linseed cake was entirely replaced by cottonseed cake and sesame cake in the case of the first and second sets of cows respectively. This

feeding was continued up to and including the 18th of April, samples being taken from each of the cows Nos. 1 to 10 daily, and from 11 to 12 at longer intervals.

Every sample from the four cows fed on cottonseed cake gave the reaction for cottonseed-oil, while the others gave negative results. Neither in the samples from the cows fed on sesame cake, nor in any of the other samples, was any indication of the sesame-oil reaction found.

As no marked increase in the intensity of the cottonseed-oil reaction was apparent at the end of the month's feeding on the normal ration of 4 lbs. of cake, the quantity, in the case of the first set of cows (Nos. 1 to 4) was gradually increased to 6 lbs., and then to 7 lbs. per day, which was found to be about the maximum amount the cows would eat. This feeding was continued till the 24th of May; but, even after more than two months' feeding with cottoncake, the results of the examination of the butter showed no decided or general increase in the cottonseed-oil reaction.

From the 24th of May also the cows numbered 7 and 8 had their sesame cake gradually replaced by cottonseed cake. Samples taken less than twenty-four hours after they had received 2 lbs. of cottoncake gave the cottonseed-oil reaction, and subsequent samples gave similar results. In the case of the samples from cows Nos. 9 and 10, taken on the 26th of April and the 3rd of May, a slight cottonseed-oil reaction was observed, although the cows were apparently receiving no cottoncake. On investigation, however, it was found that a small quantity of broken cottoncake had accidentally got mixed with the chopped straw from which the cows were being fed.

The daily allowance of sesame cake was in a similar way increased to 7 lbs. (the cows would eat no more) in the case of cows Nos. 9 and 10, but no sesame-oil reaction was obtained in any of the samples from these cows even at the end of two months.

On the 25th of May the cake feeding was discontinued and the cows turned out to grass. On the 26th of May, and about every alternate day till the 17th of June, samples were taken from the first set of cows (Nos. 1 to 4), and the results of their examination showed that the cottonseed-oil reactions persisted in a distinct form for about three days, and were distinguishable in the butter from three out of the four cows on the fifth day. At the end of a week it had disappeared from all.

In a number of samples the reactions given by the tests for cottonseed-oil were carefully compared with those given by similar butterfats to which 1, 2, and 5 per cent. of four representative samples of commercial cottonseed-oils had been added. From a comparison of the results of the tests, it was apparent that the strongest reactions from the samples from the cows fed on cottonseed cake did not much exceed that given by 1 per cent. of cottonseed-oil added to butter, and the great majority of the samples gave reactions indicating less than 1 per cent.—reactions which would, indeed, be almost obscured in butter prepared and coloured in the ordinary manner.

On several occasions the Principal of the College prepared butter in the ordinary way from the mixed milks of the cows in the respective sets. The results of the examination of samples of these butters were similar to those from the individual cows, the intensity of the cottonseed-oil reaction being intermediate between the weakest and strongest cows in the set. These butters were, however, uncoloured, and the reactions were consequently not obscured.

The individual cows seemed to vary somewhat in their ability to pass the reacting substance into the milk fat—cow No. 2 giving uniformly less intense reactions than the others.

It also appears that, whilst in the butter from cows fed on linseed oilcake there is a tendency for the analytical results to vary towards those given by margarine, the tendency in the case of sesame and cottonseed oilcake feeding is in the opposite direction.

The general conclusions to be drawn from these experiments may be summed up as follows:

1. Cows fed on cottonseed oilcake produce milk the butter-fat of which gives cottonseed-oil reactions.

2. The reactions appear when the cows receive only a small quantity of cake. They increase somewhat with continuous feeding, but apparently cannot be carried beyond a certain point, even when the amount of cake is increased to the full limit which the cows under ordinary circumstances care to eat.

3. The reacting substance passes into the milk within less than twenty-four hours after the cake-feeding begins, and continues to do so for several days after it has been dropped.

4. The reactions vary in intensity in individual cows, but do not in any case much exceed those given by 1 per cent. of cottonseed-oil mixed with butter. The presumption is, therefore, that in butter made in the ordinary way from the mixed milks of several cows, the reaction would, as a rule, be less than that due to the presence of 1 per cent. of the oil, and experiment shows that this is actually the case.

5. As feeding with cottonseed oilcake gives butter affording analytical data tending to differ from, rather than to approach to, those given by margarine, it appears to be possible in most cases to differentiate between the cottonseed-oil reaction due to feeding on cottonseed oilcake and that produced by any considerable admixture of margarine containing cottonseed-oil with butter.

6. The butter from the milk of cows fed on sesame oilcake gives no sesame-oil reaction, even after more than two months continuous feeding up to as large a quantity as the cows will take.

Similar results have been obtained by other observers.

Dr. Stein (*Tiddskrift for Landökonomi*, 1894, 664) came to similar conclusions both in his cottonseed and sesame oilcake feeding experiments. Two cows only were employed.

In Dr. Werenskjold's experiments (private communication), made on the mixed milk of two groups of four cows each, the cottonseed-oil reaction was apparent.

Harrington and Adriance (*U.S.A. Exp. Station Record*, vol. vi., 323) obtained similar results from two cows fed on cottonseed-meal.

Recent experiments made by E. Ramm and W. Mintrop, at the Poppelsdorf Agricultural Academy, under the instructions of the German Imperial Health Department (*Milchzeitung*, No. 17, April 23, 1898), with sesame cake and sesame-oil feeding gave entirely negative results with the sesame-oil test.

Dr. M. Siegfeld (*Milchzeitung*, No. 21, May 2, 1898) in a similar series of experiments obtained some positive results. With one exception, however, the intensity of

the reactions was very weak—equivalent to only a few hundredths per cent. of sesame-oil; and as similar reactions were given by some butters from cows not fed on sesame cake, the results cannot be considered as very trustworthy. Dr. Siegfeld makes a point of heating the mixture during the test, which is certainly desirable; but great care is necessary, otherwise a pinkish colour may be produced which might be mistaken for the sesame-oil reaction.

APPENDIX A.—PARTICULARS OF COWS.

No.	Breed.	Age.	Date of last calving.	Remarks.
		Years.		
1	Shorthorn.	8	18th October, 1897.	Approximate age.
2	,,	,,	23rd December, 1897.	,,
3	,,	5–6	Early February, 1898.	,,
4	,,	,,	,,	,,
5	,,	,,	18th July, 1897.	,,
6	,,	,,	26th November, 1897.	,,
7	,,	,,	Early February, 1898.	,,
8	,,	,,	,,	,,
9	,,	8	,,	,,
10	,,	5	,,	,,
11	,,	5–6	19th February, 1898.	,,
12	,,	3	,,	Heifer first calf.

APPENDIX B.—PARTICULARS OF FEEDING.

Cows.	Periods.					
	I.	II.	III.	IV.	V.	VI.
No.	Lbs.	Lbs.	Lbs.	Lbs.	Lbs.	
1	4 linseed cake.	2 linseed, 2 cotton cake.	4 cotton cake.	6 cotton cake.	7 cotton cake.	Grass.
2	,,	,,	,,	,,	,,	,,
3	,,	,,	,,	,,	,,	,,
4	,,	,,	,,	,,	,,	,,
5	,,	4 linseed cake.	4 linseed cake.	4 linseed cake.	4 linseed cake.	,,
6	,,	,,	,,	,,	,,	,,
7	,,	2 linseed, 2 sesame cake.	4 sesame cake.	6 cotton cake, gradually 2 each week.	7 cotton cake.	,,
8	,,	,,	,,	,,	,,	,,
9	,,	,,	,,	6 sesame cake.	7 sesame cake.	,,
10	,,	,,	,,	,,	,,	,,
11	,,	4 linseed cake.	4 linseed cake.	4 linseed cake.	4 linseed cake.	,,
12	,,	,,	,,	,,	,,	,,

During the whole of Periods I. to V. all the cows had daily, in addition to the

cake, a ration consisting of 4 lbs. dried grains, 2 lbs. bean meal, 25 lbs. hay and chopped straw, and 40 lbs. root (swedes and mangolds).

The bean-meal and part of the grains were withdrawn from cows 1 to 4 and 7 to 10 towards the end of Period IV.

ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

FOODS AND DRUGS ANALYSIS.

Gottlieb's Method for estimating Fat in Milk. **M. Weibull.** (*Chem. Zeit.*, 1898, xxii., 632.)—The apparatus in which this process is carried out consists of a glass tube about 40 cm. long, sealed at one end, and provided with marks at the points where it holds 70·5 c.c. and 19·0 c.c., or else graduated regularly between the 16 and 20 c.c. positions. Into it is measured exactly 10 c.c. of milk (equal to 10·27 grammes), 1 c.c. of 20 per cent. ammonia is run in from a burette, and then 10 c.c. of 95 per cent. alcohol. The liquid is shaken, 25 c.c. of ether introduced, the tube closed with a damp cork, and inverted several times. Twenty-five c.c. of petroleum spirit are next added to remove water, sugar, etc., from the ether, and after the whole has been thoroughly agitated, it is allowed to rest until the liquids have separated perfectly. In the case of milks poor in fat, the line of division is exactly at the 17·5 c.c. point (when there is one); then the ethereal liquid is syphoned off down to the 19 c.c. mark (leaving 1·5 c.c. behind, and yielding a solution containing the fat in 10 grammes of milk), evaporated or distilled, dried for two hours at 100° C., and weighed. The petroleum spirit should boil below 80°, and both it and the ether must be free from fixed residue, but the latter need not be dry. If the original sample is sour, it is treated with exactly 10 per cent. of ammonia, and 11 c.c. (equal to 10 c.c. of milk) are measured off. Buttermilk sometimes contains membranous substances which adhere to the walls of the tube and pass through the syphon, contaminating the fat solution. This may be avoided by the addition of 10 per cent. of ammonia as before, taking 10 c.c. for analysis, and adding 10 per cent. to the figures obtained. Provided the recovered fat forms a perfectly clear oil when taken out of the drying oven, and that it is soluble in a very small volume of dry ether, it must be pure and free from foreign matter; and accordingly, if Gottlieb's process gives higher results than other methods, this is simply due to its superior accuracy.

In order to compare the kaolin and paper gravimetric processes with the present one, four specimens of milk were prepared and submitted to ten different analysts. Nos. I. and II. of these samples were separated milk; III. a full milk, all containing a trace of formalin; IV. was a buttermilk mixed with 10 per cent. of ammonia to ensure uniformity of composition. The subjoined table shows the average amount of fat contained in each material, as deduced from the single results of the various investigators, and as returned by the three different processes; it also records the

greatest difference from the mean found by any individual observer; while as a few of the tests were admittedly bad, the last column gives the corrected differences from the mean after the worst results have been rejected:

		Average Percentage of Fat.	Maximum Difference.	Corrected Difference
Kaolin process	I. ...	0·109	±0·036	±0·024
	II. ...	0·240	0·050	0·030
	III. ...	3·562	0·038	0·038
	IV. ...	0·340	0·070	0·028
Paper process	I. ...	0·083	0·023	0·023
	II. ...	0·239	0·021	0·021
	III. ...	3·542	0·082	0·082
	IV. ...	0·268	0·038	0·088
Gottlieb's process	I. ...	0·115	0·010	0·010
	II. ...	0·263	0·027	0·010
	III. ...	3·533	0·043	0·043
	IV. ...	0·350	0·022	0·022

Summing up the whole case, it would appear that with skim milk and buttermilk Gottlieb's process yields the most concordant results; but for the examination of full milk there is nothing to be chosen between the three methods. And although the investigation has not yet proceeded far enough to prove definitely that the figures given by the former are nearest the truth, nevertheless its greater uniformity in the hands of different chemists strongly points in that direction. The comparative failure of the process in the analysis of milks rich in fat simply depends on the fact that the line of division between the ethereal and aqueous liquids in the tube does not stand quite at the 17·5 c.c. point—because the percentage of fat being higher, that of the milk-serum is smaller—so that the volume of fat solution withdrawn does not correspond exactly to 10 grammes of milk. This may be overcome by graduating the whole of the tube, noting the volumes after separation, and evaporating the proper quantity of the ethereal liquid. Gottlieb's method is certainly simpler and cheaper to carry out than either of the others. F. H. L.

The Calculation of the Calories of Combustion in Oxygen of Cereals Calculated from Analytical Data. H. W. Wiley and W. D. Bigelow. (*Journ. Amer. Chem. Soc.*, 1898, xx., 304-316.)—As the determination of the heat of combustion is now often made in the analysis of food products, and especially when information is required as to their dietetic value, the authors have made experiments to determine to what extent the calorimetrical results, as determined by the bomb calorimeter, agree with those calculated from the different constituents of the food.

The three main constituents in cereals are carbohydrates, proteids, and oils, while there are smaller amounts of other substances, such as amides, organic acids, lecithins, and colouring matter, which collectively evolve a considerable quantity of heat on combustion.

The different classes of compounds grouped under the term of carbohydrates show considerable variations in calorific power. In round numbers the heat value of the pentoses (arabinose, xylose), and of lactose (crystallized), dextrose, and fructose, is 3,750; of sucrose, maltose, and lactose (anhydrous), 3,950; and of starch

and cellulose 4,200 calories per gramme. The value of the pentosans is about 50 calories greater than that of the pentoses, and their factor is therefore 3,750 + 50 = 3,800.

In the absence of more reliable information on the results of the combustion of different proteid substances, the authors have adopted the factor 5,900 calories per gramme as being the one most generally correct. They consider it the best to determine the non-proteid nitrogen and to use this factor for the residual proteids. Asparagine may be taken as a representative vegetable amide, and the factor for asparagine is 3,400 calories per gramme.

The following example is given of the use of these data in the computation of the heat value of the nitrogenous constituents of cereals: A sample of wheat-flour was found to contain 10·71 per cent. of proteids and 0·48 per cent. of amide bodies calculated as asparagine. The calories of combustion were calculated as follows:

	Calories.
0·1071 gramme proteids × 5,900 = ...	631·9
0·0048 gramme asparagine × 3,400 = ...	16·3
Total calories due to nitrogen compounds ...	648·2

The oils extracted from different cereals and purified in the usual manner gave the following results on combustion in calories per gramme: Wheat-oil, 9,359; rye-oil, 9,322; maize-oil, 9,280. The nearest round numbers for these would be 9,350 for the wheat-oil and 9,300 for the other two. But since in calculating from the analytical data the ethereal extract contains other substances beside oil, the authors have determined the heat of combustion per gramme of the extract of several cereals. In round numbers these were: Wheat, 9,100; oats, 8,950; barley, 9,100; and rye, 9,200. In order to make allowance for the small amounts of colouring matter, organic acids, and other substances not included in the foregoing data, they propose to use the general factor 9,300 for the multiplication of the ethereal extract, both of flours and meals and of baked products.

The following tables give the calorific results calculated from the analysis in comparison with those determined by direct combustion:

Chemical Composition of Samples of Grain.

	Wheat, No. 1. Per Cent.	Wheat, No. 2. Per Cent.	Rye. Per Cent	Unhulled Oats. Per Cent.	Hulled Barley. Per Cent.
Moisture	11·33	10·65	11·71	9·26	12·20
Ash	1·69	1·77	2·31	3·78	0·93
Ether extract ...	2·00	2·24	1·63	4·72	0·92
Proteids	12·19	14·44	11·69	9·63	10·44
Sucrose	0·33	0·48	0·42	0·17	0·18
Invert sugar ...	0·027	0·08	0·068	0·031	0·007
Galactin and dextrin	0·160	0·25	0·220	0·260	0·140
Pentosan	5·80	5·17	8·10	13·65	6·50
Fibre	2·15	2·56	2·36	12·81	0·80
Starch	64·51	62·69	61·78	45·98	68·03
Calories determined by direct combustion in oxygen	3,922	4,011	3,909	4,181	3,886

CALCULATED CALORIES IN SAMPLES OF GRAIN.

Component Parts.	Wheat, No. 1. Per Cent.	Wheat, No. 2. Per Cent.	Rye. Per Cent.	Unhulled Oats. Per Cent.	Hulled Barley. Per Cent.
Ether extract ...	186	208	152	439	86
Proteids	719	852	690	568	616
Pentosans ...	220	196	308	519	247
Sucrose	13	19	17	7	7
Starch and fibre	2,800	2,741	2,694	2,469	2,891
Totals ...	3,938	4,016	3,861	4,002	3,847

The fact that the calculated calories were 179 lower than those directly determined in the case of the unhulled oats suggests the possibility that the heat of combustion of the unidentified carbohydrates of hulls and fodders is greater than that of starch. The authors consider that the direct combustion and the analysis should afford a check as to the correctness of each. At present they regard the check as satisfactory when the difference between the observed and calculated calories does not exceed 50 to 75.

In the following table of the comparative results obtained with various substances, the proteid has been calculated from the nitrogen by the factors 5·70 and 6·25:

CEREAL PRODUCTS.

	Determined Calories per gramme.	Calculated N×5·70.	Calculated N×6·25.
Flap jack flour ...	3,700	3,776	3,790
Pancake flour ...	3,820	3,833	3,845
Griddle cake flour ..	3,724	3,706	3,719
Palen flour	3,870	3,880	3,894
Mean	3,779	3,779	3,812
Breakfast and Partially-prepared Foods.			
Shredded whole-wheat biscuit ...	4,253	4,298	4,314
Wheat-germ meal ...	4,362	4,404	4,420
Gluten butter wafers	4,610	4,605	4,628
Whole-wheat gluten	4,544	4,542	4,566
Cooked gluten ...	4,432	4,406	4,425
Germea	4,445	4,403	4,417
Breakfast gem ...	4,379	4,405	4,420
Cracked wheat ...	4,453	4,395	4,413
Mean	4,435	4,432	4,450

Bread.

	Determined Calories per gramme.	Calculated N×5·70.	Calculated N×6·25.
Vienna	4,420	4,372	4,394
,, ...	4,381	4,339	4,358
Home-made ...	4,558	4,483	4,503
	4,436	4,450	4,465
Mean	4,449	4,411	4,430
Miscellaneous.			
Kaffee brod... ...	4,146	4,203	
Granula	4,385	4,399	
Imperial granum ...	4,485	4,462	
F.F.V. malt food ...	4,470	4,465	
Granulated barley ...	4,365	4,353	
H.O. oatmeal ...	4,800	4,799	
Mean	4,442	4,447	

The authors are investigating the data for the calculation of heat of combustion in bodies rich in ligno-cellulose, etc., such as bran, hay, and fibres generally, and promise a further communication on the subject. C. A. M.

Detection of Sawdust in Flour. G. A. Le Roy. (*Zeit. für Untersuch. der Nahr. und Genussmittel*, 1898, 505.)—Sawdust can be detected in common descriptions of flour by means of an alcoholic solution of phloroglucinol strongly acidified with phosphoric acid. On gently warming, the particles of sawdust acquire a strong carmine-red colour, whereas the bran particles of the cereals are at first scarcely affected at all. H. H. B. S.

A Substance other than Sugar in Fruit Juices capable of reducing Fehling's Solution. Aderhold and Heintze. (*Chem. Zeit.*, 1898, xxii., 632.)—In estimating the proportion of sugar in the juice of cucumbers by means of Fehling's solution, the authors recently obtained very different results when the liquor was expressed from the fruit in its natural condition from those yielded by the aqueous extract of the cucumbers after they had been previously dried. On adding a moderate amount of alcohol to the neutral juice, a gelatinous flocculent precipitate was produced, which appeared to be a pectin, and did not reduce Fehling's solution; but on filtering the liquid and adding more alcohol a yellowish-brown, resin-like body was thrown down, which exhibited powerful reducing properties. It was readily soluble in water, had a sharp, not a sweet taste, and entirely lost its reducing power when its aqueous solution was evaporated and the residue dried on the water-bath for several hours. This peculiarity no doubt explained the anomalous behaviour of the fresh cucumber juice; for hitherto the new body has always been returned as a sugar when Fehling's process is made use of. The same substance has been detected in strawberries, bilberries, gooseberries, and cherries, and probably exists in all fruits, in which, when ripe, the proportion is small. In green apples, however, it is considerable. F. H. L.

The Determination of Malic Acid in Grape Juice. A. Girard and Lindet. (*Bull. Soc. Chim.*, 1898, xix., 585.)—The tartaric acid (free and combined) is first determined as tartar by the method of Berthelot and Fleurien, in which the hydrogen potassium tartrate is precipitated by a mixture of alcohol and ether, and the free tartaric acid by saturating about one-third of the liquid (containing the ether) with potash, and adding the remaining two-thirds.

After removal of the tartaric acid the alcohol and ether are expelled by evaporation, and a solution of normal lead acetate added to the boiling aqueous solution until there is a permanent turbidity. The boiling liquid is filtered, and the lead malate allowed to crystallize. The next day the mother liquor is treated in the same way, the lead malate collected, and the process again repeated, and so on until no more crystals are deposited on cooling. The lead malate is then dried, detached from the filter, and weighed.

Since lead malate is soluble to some extent in acetic acid, a correction must be made at the end of the determination for the amount of free acid in the mother liquid. This can be done by means of the following table:

Quantity of free Acetic Acid in 100 c.c. of Mother Liquid.	Lead Malate dissolved in 100 c.c. of Mother Liquid.
0·21	0·10
0·40	0·13
0·55	0·16
0·73	0·18
0·90	0·20

The authors state that test experiments with this method have given satisfactory results. C. A. M.

On the Recognition of White Wines prepared by the Decolorisation of Red Wines. A. Bimm. (*Journ. Pharm. Chim.*, 1898, viii., 9-11.)—When white wine has been prepared by the decolorization of red wines by means of animal charcoal, the author finds that the sophistication can often be detected in the following manner:

A solution of diphenylamine is prepared by dissolving 0·1 gramme in 100 c.c. of sulphuric acid (1 : 4), and making up the volume to 500 c.c. with sulphuric acid of 66°. To 2 c.c. of this reagent in a porcelain dish 6 drops of the wine are added, and if a blue coloration is obtained, it points to the wine being a decolorized red wine. The author has obtained this reaction with a large number of white wines, which, from the results of the analysis, were suspected of being decolorized products. With some of these the colour was partially restored on the addition of hydrochloric acid. On the other hand, the blue coloration did not appear in the case of white wines of authentic origin and normal composition.

The substance which causes the blue colour was found to be a usual constituent of animal charcoal, which dissolves in the wine and gives the reactions of a nitrate.

C. A. M.

Studies on the Products obtained on Roasting Coffee. H. Jaeckle. (*Zeit. für Untersuch. der Nahr. und Genussmittel*, 1898, 457.)—The products were collected by means of a condenser cooled by ice attached to the coffee-roaster. The distillate was a brownish-yellow fluid having the aroma of freshly-roasted coffee and a bitter sour taste. The following products were identified, viz., acetone, furfurol, caffeine, ammonia, trimethylamine, formic acid, acetic acid (resorcin). Of these products, caffeine, furfurol, and acetic acid were found in somewhat considerable quantities; but acetone, ammonia, trimethylamine, and formic acid in only very small proportions. Bernheimer's "caffeol" could not be detected. That caffeine was found in the distillate accords with the recent observation that coffee sustains a loss of caffeine in roasting.

H. H. B. S.

A New Method of Sophisticating Coffee. T. F. Hanausek. (*Zeit. für Untersuch. der Nahr. und Genussmittel*, 1898, 399.)—The author remarks that the practice of centrifuging coffee-berries with sawdust to improve their appearance, referred to by Wirtz (*Zeit. für Untersuch. der Nahr. und Genussmittel*, 1898, 248; ANALYST, 1898, this vol., 209), has been carried on for several years. He considers it questionable that it is done with the object of imparting a fine white to the furrows, as stated by Wirtz, and believes the object is merely the removal of the mottled appearance due to the outer skin, and the imparting of more or less polish to the berries. He, therefore, disagrees with Wirtz in considering the practice as equivalent to adulteration, though he would prefer to see the sawdust completely removed. The practice of colouring coffee-berries is, however, in the author's opinion, quite another matter, and he proposes to deal fully with this in a future paper.

H. H. B. S.

The Detection of Gelatin in Chocolate. P. Onfroy. (*Journ. Pharm. Chim.*, 1898, viii., 7-9.)—By the addition of 5 per cent. of gelatin it is possible to introduce at least 10 per cent. of water into a chocolate without altering the appearance of the latter. A sample of chocolate thus adulterated gave the following percentage results on analysis: Ash, 2·72; sugar, 46·17; fat, 14·24; water, 12·20. Twenty-five grammes of this product when mixed with hot water gelatinized on cooling.

The results obtained with five samples of chocolate manufactured by reliable makers were:

		Per cent.				
Ash	...	1·07	1·46	1·36	2·21	1·69
Fat	...	21·60	19·90	21·05	19·60	20·50
Sugar	...	63·99	63·18	60·75	59·94	61·56
Water	...	0·98 to 2·50				

As a positive test for gelatin 5 grammes of the suspected chocolate are treated with 50 c.c. of boiling water and 5 c.c. of a 10 per cent. solution of lead acetate added. The liquid is filtered, and several drops of a saturated aqueous solution of picric acid added to the filtrate. In the presence of a notable quantity of gelatin a yellow precipitate is obtained.

When the gelatin is only present in traces, the tannin in the cocoa combines with it, forming an insoluble precipitate, which remains on the filter. In such cases 10 grammes of the chocolate are extracted with ether to remove the fat, the residue treated with 100 c.c. of hot water, and the liquid filtered after the addition of 5 to 10 c.c. of a 10 per cent. solution of potassium hydroxide and 10 c.c. of the lead acetate solution. The filtrate is exactly neutralized and tested for gelatin in the usual way.

These tests are only qualitative, but an approximate idea of the quantity of gelatin present can be calculated from the amounts of the other constituents.

C. A. M.

Utilization of Cacao-husks. G. Paris. (*Zeit. für Untersuch. der Nahr. und Genussmittel*, 1898, 389.)—Cacao-husks are now finding a much more extended use than was formerly the case. The roasted husks, on account of their aroma, are readily eaten by cattle, and may be advantageously given in conjunction with other foods. They are also used for the preparation of cheap beverages. Treated after the manner of tea, they yield an extract of a bitter flavour, which, on the addition of sugar and milk, is used as a substitute for coffee or chocolate.

According to the author's analysis, the following is the percentage composition of roasted husks:

Water.	Nitrogenous Matter.	Fat.	Non-nitrogenous Extractial Matter.	Crude Fibre.	Ash.
12·57	14·69	3·30	45·76	16·33	7·35

A decoction of 50 grammes of the powdered roasted husks in hot water diluted after cooling to 500 c.c. yielded the following results upon analysis:

		Per Cent.				
Sp. Gr. at 15° C.	Extract.	Organic Matter.	Ash.	Reducing Sugar (Dextrose).	Theobromine.	Acid (Tartaric).
1·1269	25·08	20·68	4·40	0·21	0·79	0·12

The presence of cacao-husks in cocoa-powder may in most cases be detected microscopically by the annular, spiral, and small hexagonal cells of the husks and the characteristic table-shaped cells of the epidermis. H. H. B. S.

A New Pepper Adulterant. T. F. Hanausek. (*Zeit. für Untersuch. der Nahr. und Genussmittel*, 1898, 490.)—A sample of ground pepper from Prague was found to contain foreign particles not corresponding with any of the substances usually employed for mixing with pepper. Three special peculiarities were observable: (1) bundles of corrugated bent fibrous cells; (2) coarse parenchyma overlaid with narrow cells of a yellow colour with parallel walls; and (3) colourless cellular parenchyma, firm in the walls and enclosing numerous beautiful crystal rosettes and granules. The two last peculiarities were recognised as characteristic of a fruit of the order Umbelliferæ, the bundles of fibres as well as the absence of vittæ (oil cavities) pointed to coriander, and a comparison with the structure of this fruit showed without doubt that the pepper had been adulterated with ground coriander-seeds. H. H. B. S.

Adulteration of Vanillin in Switzerland. R. Hefelmann. (*Apoth. Zeit.*, 1898, xiii., 420; through *Chem. Zeit. Rep.*, 1898, 181.)—A sample of vanillin supplied by Ewald and Börlin, of Basle, was recently examined, and found to contain about 26 per cent. of acetanilide. F. H. L.

Physiological Action of Copper. Baum and Seeliger. (*Zeit. für Untersuch. der Nahr. und Genussmittel*, 1898, 500; from *Zeit. öffent. Chem.*, 1898, 181.)—The authors have carried out a large number of experiments upon animals extending over a period of three years. The following are some of the principal results arrived at:

Copper absorbed into the system through the digestive organs does not, as a rule, pass into the milk. Occasionally it is found in minute traces in the milk, but only quite exceptionally in weighable amounts. No injurious consequences could be traced to the use of milk produced by animals that had been dosed for long periods with copper, not even when the milk was used as an exclusive diet, as, for example, or suckling.

Copper absorbed into the system through the digestive organs passes in proportionate quantity into, and is stored in, the fœtus.

When administered in small daily doses, copper is, as a rule, all absorbed, and for the most part all again eliminated. When increased quantities are given, however, no more is absorbed—often, indeed, less. The absorption and elimination, therefore, does not appear to proceed with regularity or to follow any decided rule.

The elimination of the copper through the bile and pancreatic and intestinal juices may take as much as five months from the time the administration was discontinued, though it may be complete in four or five weeks. By long-continued administration of small doses of copper a really chronic poisoning may be produced.

Different copper preparations possess poisoning properties in different degrees.

H. H. B. S.

Contribution to our Knowledge of Pilocarpidine. J. Herzig and H. Meyer. (*Monatshefte für Chemie*, 1898, xix., 56.)—The authors discuss the subject of the assumed identity of Merck's pilocarpidine from jaborandi-leaves with the supposed pilocarpidine of Hardy and Calmels, Petit and Polonowski, produced from pilocarpine. They had already pointed out (*Monatshefte für Chemie*, xviii., 382) that the evidence of identity was insufficient, and they now show by means of the determination of methyl that they are able to completely corroborate Merck's conclusion, arrived at in a different way, thus placing the non-identity of these bodies beyond question. As additional proof, the authors have made methyl determinations in the double salts of gold of these bodies, and give the following results:

PILOCARPIDINE DOUBLE SALT WITH GOLD.

	Found.		Calculated for $C_{10}H_{14}N_2O_2$, HCl, $AuCl_3$
	I.	II.	
Au	36·75	—	36·85
CH_3—N	—	0·19	2·81

CONVERTED PILOCARPINE DOUBLE SALT WITH GOLD.

	Found.		Calculated for $C_{11}H_{16}N_2O_2$, HCl, $AuCl_3$
	I.	II.	
Au	35·85	—	35·72
CH_3—N	—	3·07	2·74

H. H. B. S.

Contributions to our Knowledge of the Alkaloids of Macleya Cordata. K. Hopfgartner. (*Monatshefte für Chemie*, xix., 179.)

ALKALOID I. (*Macleyine, Protopine*).

The melting-point of the free base was given by Eykman as 201° C. Selle, however, gave the melting-point of a base obtained from *Chelidonium majus*, the identity of which with macleyine is in the highest degree probable, as 207° C. The author at first found the melting-point of the free base to be 202° to 203° C., but subsequent determinations, after repeated crystallization from alcohol, gave 207° C. The following is supplementary to Eykman's description of this alkaloid: The base is difficultly soluble in carbon bisulphide and benzene, and very difficultly soluble in petroleum spirit. It crystallizes best from a mixture of alcohol and chloroform, in colourless, lustrous, four-sided prisms. The reactions with sulphuric acid were found to agree with the description given by Eykman. Zeisel's method for the determination of methoxyl gave negative results. Determinations of carbon and hydrogen corresponded with the formula $C_2H_{19}NO_5$. The base is optically inactive. The nitrate is a white powder which is seen to be crystalline when observed under the microscope. It is difficultly soluble in cold water, but more so in hot, from which it crystallizes out on cooling. Determinations of carbon and hydrogen corresponded with the formula $C_{20}H_{19}NO_5$, HNO_3.

The hydrochloride separates out in six-sided prisms on neutralizing the base with

hot dilute hydrochloric acid and cooling. It is soluble in alcohol and with some difficulty in cold water. Its analysis corresponded with the formula

$$C_{20}H_{19}NO_5, HCl + \tfrac{1}{4}H_2O.$$

ALKALOID II.

This is obtained in the form of nitrate from the mother-liquor of the protopine nitrate crystallization, from which the base is precipitated by potash as a white amorphous powder, which after long standing under the fluid becomes gradually crystalline. The crystallized base dissolves easily in chloroform, less easily in alcohol, carbon bisulphide, benzene, and acetic ether, and still less in ordinary ether. Its composition agrees with the formula $C_{21}H_{23}NO_5$. Zeisel's method revealed the presence of two methoxyl groups. The base is optically inactive. The nitrate of base II. is a colourless salt, soluble in alcohol, and moderately difficultly soluble in cold water. Determinations of carbon, hydrogen, and water agreed with the formula

$$C_{21}H_{23}NO_5, HNO_3 + 1\tfrac{1}{2}H_2O.$$

Dried at 100° C., its formula is

$$C_{21}H_{23}NO_5, HNO_3 + \tfrac{1}{4}H_2O.$$

The hydrochloride crystallizes out in the form of fine white needles on cooling a solution of the free base in hot dilute hydrochloric acid. Its composition corresponds to the formula

$$C_{21}H_{23}NO_5, HCl + 1\tfrac{1}{2}H_2O.$$

Dried at 100° C., its formula is

$$C_{21}H_{23}NO_5, HCl + \tfrac{1}{2}H_2O.$$

There is also a double salt of platinum of the formula

$$(C_{21}H_{23}NO_5, HCl)_2PtCl_4.$$

H. H. B. S.

Oil of Cade. P. Adam. (*Bull. Soc. Chim.*, 1898, xix., 580-583.)—This oil is largely employed as a therapeutic agent in certain diseases of the skin. The pure product has been examined by Hirschsohn (*Pharm. Zeit. f. Russ.*, 1895, xxxiv., 817) and by De Schulz (*Arch. de Sc. Viol. St. Petersburg*, 1897, 345-374), and the author's researches have been carried out with the sole purpose of determining whether it was possible for a pharmacist to obtain an oil answering to the requirements of the French Codex.

The different specimens examined were supplied by reliable French firms, and were tested in accordance with Schulz's directions. On treatment with caustic potash all absorbed oxygen with great rapidity from the air. They were fractionated by distilling 300 grammes under the ordinary pressure, and each fraction was dissolved in ether and shaken with a solution of an alkaline carbonate to remove the free acids. The ethereal layer was then shaken with a solution of caustic potash, which dissolved the phenols and polyphenols, leaving the phenol esters, ketones, and other neutral bodies. In each case different results were obtained.

All the samples were completely soluble in amylic alcohol, ether, benzene, chloroform, carbon bisulphide, and aniline, but gave a more or less turbid solution

with olive-oil, in which, according to De Schulz, the pure oil is completely soluble. Turpentine should also dissolve the oil completely, but in none of the specimens examined by the author was there complete solution.

The results of other tests are given in the subjoined table, in which the names at the top of the columns are those of the firms which supplied the oil:

	Characteristics given by De Schulz.	Pelliot.	Poulenc.	Pharmacie Centrale.	P. Bounes.	Cabanis.
Density	0·9874 to 0 9904	1 at 20° C.	0·998 at 18·5° C.	1·031 at 14° C.	1·02 at 14° C.	0·998 at 25° C.
Viscosity (minutes)	?	105	60	270	360	32
Solubility in Alcohol 90 per cent.	Incomplete	Complete.	Complete	Turbid	Turbid	Turbid
Solubility in Acetic acid	Incomplete	,,	,,	Complete	Complete	Complete
Colour reaction of solution in ether (1 : 10) with copper acetate (1 : 1000)	Greenish	Nil	Nil	Nil	Red	Nil
Colour reaction of aqueous extract (1 : 10) with ferric chloride (1 : 1000)	Red	Vinous red	Onion-peel colour	Vinous red	Onion-peel colour	Onion-peel colour
Lime-water	Reddish-brown	Colour hardly perceptible	Nil	Yellow	Yellow	Onion-peel colour
Ammonia	Madder	Yellow	Madder	,,	,,	Yellow
Per cent. distilling below 250° C.	—	12	7	11	9	9
Between 250° and 275° C.	—	33	36	12	10	29
Between 275° and 300° C.	—	26	34	14	10	37
Above 300° C. and residue	—	30	27	65	72	28
		101	104	102	101	103

The fact that in every instance the total amount obtained on distillation was greater than the quantity taken is attributed to the rapid oxidation of the oil. The author considers that these results show that it is practically impossible to obtain a pure oil of cade, and that therefore that oil ought to be omitted from the Codex.

C. A. M.

ORGANIC ANALYSIS.

Detection of Formaldehyde. D. Vitali. (*Boll. Chim. Farm.*, 1898, xxxvii., 321; through *Chem. Zeit. Rep.*, 1898, 179.)—When an aqueous solution of formaldehyde is treated with a 4 per cent. solution of a salt of phenylhydrazine, a white precipitate is produced, and the mixture gradually turns yellow and then orange. The reaction is given instantly by 1 per cent. solutions; in 0·001 per cent. solutions it requires two or three hours to develop. The deposit is amorphous and dissolves in alcohol, crystallizing therefrom in rhombic tables. If the mixture of formaldehyde

and phenylhydrazine is allowed to evaporate spontaneously, a yellow residue is obtained, which finally becomes red; and on washing this with water, dissolving it in warm absolute alcohol, a yellow solution is produced which reduces gold salts, giving a green liquid that changes to red on addition of caustic soda. If the original mixture, immediately the precipitate falls, is treated with sodium nitroprusside and caustic soda, it yields a fine blue colour. Acetaldehyde does not give the reaction; and provided the latter is not present in too large proportions, it may be used for the recognition of formaldehyde therein. F. H. L.

Electrolytic Determination of Sugar. J. Formánek. (*Zeit. für Untersuch. der Nahr. und Genussmittel*, 1898, 320.)—This is substantially Allihn's method, but modified so as to render it easier of execution. The original process is followed exactly up to the point of the separation of the cuprous oxide. This is then filtered off through a good thick Swedish filter, which has been successively extracted by hydrochloric and hydrofluoric acids. The precipitate must be kept covered with liquid as completely as possible during the filtration and subsequent washing. It is washed with hot water and then dissolved in the smallest possible quantity of warm dilute nitric acid (specific gravity, 1·2), the acid solution and washings being collected in a weighed platinum basin. Ammonia is now cautiously added until a faint blue colour is produced, after which, for quantities of copper up to 0·5 gramme, 20 c.c. of ammonia of 0·96 specific gravity, and 20 c.c. of a 25 per cent. solution of ammonium nitrate, are added, and the solution diluted to about 150 c.c. For quantities of copper exceeding 0·5 gramme, 30 to 35 c.c. of ammonia must be used. On applying the current, the deposition of copper proceeds very rapidly, and is complete as soon as the last shade of blue disappears from the liquid. The basin is then washed out, at first without interrupting the current, and finally rinsed with distilled water and absolute alcohol. It is then dried for five minutes at 80° to 90° C., cooled and weighed. From the increase in weight, corresponding to the amount of copper, the sugar is found by reference to the tables.

The following results are given:

No.	Copper found.	Glucose found.	Glucose taken.	Difference.
1.	0·0948	0·0483	0·04865	− 0·00035
2.	0·0956	0·0487	0·04865	+ 0·00005
3.	0·0958	0·0488	0·04865	+ 0·00015
4.	0·1896	0·0971	0·0973	- 0·00020
5.	0·1901	0·09735	0·0973	+ 0·00005
6.	0·1906	0·0976	0·0973	+ 0·0003
7.	0·1896	0·0971	0·0973	- 0·0002
8.	0·1900	0·0973	0·0973	0
9.	0·1896	0·0971	0·0973	− 0·0002
10.	0·2790	0·1450	0·1459	− 0·0009
11.	0·2800	0·1455	0·1459	− 0·0004
12.	0·2795	0·1453	0·1459	− 0·0006

H. H. B. S.

Sugar Estimation according to Kjeldahl. Bruhns. (*Centralbl. Zuckerind.*, 1898, vi., 755; through *Chem. Zeit. Rep.*, 1898, 180.)—Kjeldahl's criticisms (ANALYST, xx., 227) on the processes for estimating sugar with alkaline copper solutions are no doubt well-founded when the conditions are such as those detailed in his experiments; but these conditions do not exist, or can easily be avoided, in practice. Access of oxygen to the surface of the liquid, even under the most unfavourable circumstances, does not appreciably affect the results of the analysis. F. H. L.

A Method for the Differentiation of Organic Matter in Water. A. G. Woodman. (*Journ. Amer. Chem. Soc.*, 1898, xx., 497-501.)—The author's objections to the Forchammer permanganate process and its modifications are: (1) That the results are comparative, and do not indicate the absolute amount of carbonaceous matter, and (2) that these comparative results are only of value when the organic matter in different samples is identical in composition.

Barnes (*Journ. Soc. Chem. Ind.*, 1896, 83) showed that organic matter underwent different degrees of oxidation with permanganate and chromic acid, and expressed the results in the form of a ratio between the amount of oxygen consumed in each case. For example, solutions of starch and of sugar are acted upon by chromic acid much more than by permanganate, thus giving a very low ratio. Gelatin and glycerin also give low ratios, whilst, on the other hand, infusions of peat and solutions of tannin give a high ratio. On these observations the author has based his method of determining the nature of the organic matter in water.

One hundred c.c., or more, of the water are mixed with 8 c.c. of dilute sulphuric acid (1 : 3), and about 10 c.c. of $\frac{N}{100}$ potassium permanganate (or more if the water was highly coloured) added from a burette. The liquid is heated to boiling, boiled for exactly five minutes, allowed to cool for one minute, 10 c.c. of $\frac{N}{100}$ oxalic acid added, and the excess of the latter titrated with the standard permanganate.

The solutions required for the chromic acid method are: (*a*) 6·2 grammes of potassium bichromate dissolved in water, and after the addition of 50 c.c. of concentrated sulphuric acid made up to a litre; (*b*) 18 grammes of crystallized ferrous sulphate dissolved in a mixture of 100 c.c. of sulphuric acid and 200 c.c. of water, and diluted to 500 c.c. This solution will not keep for more than two or three weeks.

Fifty c.c. of the water to be examined are mixed with 20 c.c. of sulphuric acid (specific gravity 1·84), and after the addition of exactly 10 c.c. of the chromic acid solution, heated for an hour on the boiling water-bath in a flask covered with a watch-glass. It is then allowed to cool for a few minutes, 10 c.c. of the ferrous sulphate solution added, and the whole poured into 500 c.c. of cold water and titrated with $\frac{N}{100}$ permanganate. A blank determination should always be made at the same time on 50 c.c. of water free from organic matter.

The results are expressed in the form of an "oxygen ratio" by dividing the number of c.c. of permanganate used in the first determination by the number required by an equal volume of the water in the chromic acid method. In general this ratio is in the form of a fraction less than unity.

In the investigation the author applied this process to the examination of natural waters which consumed a large amount of oxygen, but which were known to be free from sewage contamination, and also to filtered city sewage in various degrees of dilution. The results obtained with these "peaty" waters are given below, the colour being those of Leed's nesslerized ammonia scale.

	Colour.	Ratio.
Boston tap-water (July 24, 1897)	0·3	0·656
,, ,, (January 24, 1898) ...	0·7	0·646
,, ,, (March 11, 1898) ...	0·48	0·649
Colour standard (*a*)	5·00	0·833
,, ,, (*b*)	1·8	0·689
Reservoir Orange, Mass.	0·58	0·659
,, Athol ,, (full of algæ) ...	—	1·132
,, ,, ,, (filtered)	1·7	0·750
Infusion of dried leaves	about 10·0	0·656
Solution of humus in ammonia	—	0·692

Of these samples, the author remarks that the Boston tap-water is a typical brown surface water, and that the colour standards (*a*) and (*b*) were very dark-coloured swamp waters diluted.

The ratios obtained with sewage were about the same as those given by Barnes for nitrogenous substances:

	Ratio.
Sewage from Marlboro (filtered, old)	0·543
,, ,, Boston (decanted, fresh)	0·389
(*a*) Filtered sewage, Hereford Street, Boston	0·470
(*b*) Sewage (*a*) diluted with 5 vols. of distilled water ...	0·490
(*c*) ,, (*a*) ,, 10 ,, ,, ...	0·460
Well contaminated with sewage	0·449

From these results it is seen that peaty waters give a ratio above 0·600, whilst sewage and waters contaminated by sewage give a ratio below 0·600, which appears to be the dividing-line between the ratios of animal and vegetable origin. Moreover, the ratio is practically constant if the organic matter does not alter in character, however much the amount present may vary.

As a further test of the applicability of the process, two samples of water were prepared: (*a*) Containing considerable colouring matter of vegetable origin, but known to be free from sewage; (*b*) consisting of distilled water contaminated with sewage. The amount of oxygen consumed in the permanganate process by these samples was nearly identical ($a = 0{\cdot}2387$ and $b = 0{\cdot}2375$ parts per 100,000), but the ratios differed widely—$a = 0{\cdot}827$, and $b = 0{\cdot}372$.

The method was also tried on a well-water which appeared suspicious from the amount of oxygen consumed in the permanganate test. The ratio obtained was high —viz., 0·957—and from this the author concluded that infiltration of surface-water carrying vegetable substances, and not sewage, was the cause of the organic matter, a conclusion which was subsequently confirmed by an examination of the well.

C. A. M.

Detection of Cotton, Sesamé, and Arachis Oils in Olive-oil. M. Tortelli and R. Ruggeri. (*Chem. Zeit.*, 1898, xxii., 600.)—The authors find that by a still further modification of the process they have already described (ANALYST, xxiii., 179) for the detection of cotton-oil in olive-oil, they are able in one operation to discover the presence of sesamé and arachis, and even to determine the proportion of the latter. Twenty grammes of the sample are saponified with 50 c.c. of alcoholic potash (120 grammes in 1 litre of 90 per cent. spirit) on the water-bath; a few drops of phenolphthalein are added, and the liquid is neutralized with 10 per cent. acetic acid. Two hundred c.c. of a 10 per cent. solution of lead acetate and 100 c.c. of water are then heated to the boil in a wide-necked flask, the soap solution is run in gradually, and the whole is shaken for ten minutes in boiling water. When the lead soaps have all adhered to the walls of the flask, the clear liquor is poured off, the insoluble matter washed three times with 200 c.c. of water at 70° or 80°, cooled, dried with filter-paper, and extracted for twenty minutes with 220 c.c. of redistilled ether under a reflux condenser, with occasional agitation. The flask is next placed in running water for half an hour to allow the insoluble lead salts of the solid fatty acids to settle; and then the ethereal liquid, with as little of the precipitate as possible, is run through a filter into a separatory funnel, and the residue extracted as before with 100 c.c. of warm ether. The filtrate is decomposed with 150 c.c. of 20 per cent. hydrochloric acid, the aqueous portion drawn off, and another, or even a third, 100 c.c. of acid are added till the whole of the lead chloride is removed. The ether is washed twice with 150 c.c. of water, filtered, and distilled until 15 or 18 c.c. of liquid acids are obtained. Five or six c.c. of this residue are tested with silver nitrate for cotton-oil as previously described (*loc. cit.*), while the remainder is shaken with an equal volume of strong HCl and 2 drops of a solution of furfuraldehyde (1 gramme in 100 c.c. of 95 per cent. alcohol). In the presence of even 1 per cent. of sesamé-oil, the acid becomes red, the depth of colour being in proportion to the amount of the impurity. Under these conditions, both the Becchi test for the cotton-oil and the Baudoin test for the sesamé-oil are absolutely characteristic, since neither reaction is given by any other edible oil that may exist in the original specimen.

The rest of the insoluble lead soaps are then brought on to the filter, and the flask and precipitate washed with ether till all soluble matter is removed. The paper is pierced, its contents rinsed into a separatory funnel by means of ether, the liquid is made up to about 220 c.c., the fatty acids are liberated with 20 per cent. HCl as above, washed with water, filtered, and the solvent completely distilled off. One hundred c.c. of 90 per cent. alcohol and 1 drop of weak acid are added, the flask is closed with a cork bearing a thermometer, and the whole is shaken on the water-bath until the temperature reaches about 60° C. and a clear solution is produced. The flask is then placed on a sheet of white paper, and if the oil contains even less than 5 per cent. of arachis, as the liquid cools very fine glittering needles of lignoceric acid are deposited, which soon collect into clusters, together with a larger quantity of thin mother-of-pearl-like plates of arachidic acid. With the solitary exception of cotton-oil, when treated in this manner, other oils give no precipitate, at least during several hours' standing; and the deposit from cotton-oil cannot be

mistaken for that from arachis, as the former is amorphous and quite opaque. The temperature at which the crystals begin to appear depends on the proportion of arachis present, so that the thermometer affords a rough means of estimating the amount of impurity, as shown by the following table :

First crystals appear C°	35–38	31–33	28–30	25–26	22–24	20·5–21·5	18–20	16–17
Percentage of arachis ...	100	60	50	40	30	20	10	5

For a more accurate determination of the arachis oil, the liquid is allowed to rest at 15° or 20° for three hours, the crystals are collected on a filter, washed three times with 10 c.c. of 90 per cent. alcohol, then with 70 per cent., and dissolved in boiling absolute spirit. The solvent is distilled off, the residue mixed with 100 c.c. of 90 per cent. alcohol, heated to 60° with a drop of very weak HCl, and crystallized once more. (This recrystallization is only necessary in the presence of cotton-oil, which also contains a solid fatty acid but slightly soluble in cold 90 per cent. spirit; nevertheless, as the process is not much trouble, it is safer always to employ it, in order to ensure the absolute purity of the arachis acids.) After three hours the crystals are filtered, washed three times with 10 c.c. of 90 per cent., then completely with 70 per cent. alcohol, dissolved in absolute alcohol, evaporated in a tared basin, dried at 100° C. for one hour, weighed, and their melting-point observed. As the solubility of the two acids is different, and depends on the temperature of the liquid, the amount given in the annexed table must be added to the actual weights; and then, on multiplying by $\frac{5}{0{\cdot}048}$, the percentage of earthnut-oil in the 20 grammes of olive-oil originally taken will be obtained :

Weight of Mixed Arachis Acids.	Ten perature of 90 per cent. Alcohol. 15°.	17·5°.	20°.
Between 2·70 and 0·50 gramme ...	0·070	0·080	0·090
,, 0·47 and 0·17 ,, ...	0·050	0·060	0·070
Less than 0·11 ,, ...	0·033	0·040	0·045

In the concluding portion of their article, the authors discuss the figures quoted by previous investigators (especially Renard) regarding the solubility of "arachidic acid" in 90 per cent. alcohol; they confirm Kreiling's statement that the crystals deposited from spirit of this strength really consist of two acids of the series $C_nH_{2n}O_2$ —true arachidic acid, $C_{20}H_{40}O_2$, and lignoceric acid, $C_{24}H_{48}O_2$; and they append a table giving the melting-points and solubilities of the mixed acids at various temperatures. As the result of a number of tests on arachis-oil coming from different localities, they find that the proportion of these acids always ranges between 4·30 and 5·40 (mean 4·80) per cent., and the melting-point of the mixture lies between 74° and 75° C. They also record the effect of their process when applied to olive-oil containing sesamé, rape, cotton, and maize oils, as well as the different proportions of arachis mentioned in the first table reproduced above, proving that the latter may be determined to within 2 per cent. of the amount actually present. F. H. L.

Estimation of Arachis in Oil-Mixtures. F. Jean. (*Rev. Chim. Ind.*, 1898, ix., 162; *Les Corps Gras*, 1898, xxiv., 353; through *Chem. Zeit. Rep.*, 1898, 179.)—Ten grammes of the sample are heated to 110° C. in an enamelled iron dish, 3 grammes of caustic potash dissolved in 3 or 4 c.c. of water and 5 c.c. of alcohol (36%) are added, and the mixture is stirred with a metal spatula till the soap is dry. After cooling, it is mixed in a flask with 100 c.c. of alcohol which has previously been saturated at 11° or 12° C. with the potassium salt of arachidic acid. The mass is heated under an inverted condenser, then cooled and set aside at 15° for twelve hours. The insoluble matter is filtered off, dried by pressure in paper, placed in the flask again, and once more treated with saturated alcohol of 36% exactly as before. When it has stood for another twelve hours, the potassium arachidate is filtered, pressed in paper till dry, and decomposed with 50 c.c. of boiling weak hydrochloric acid. The liberated arachidic acid is dissolved in about 10 c.c. of petroleum spirit, washed with warm water, evaporated at 100°, and weighed. Edible arachis-oil contains as an average 4·55 per cent. of arachidic acid, and its melting-point should be at least 72° C.

(See also preceding abstract.) F. H. L.

A Method of analysing Oxidized Oils. W. Fahrion. (*Zeit. angew. Chem.*, 1898, 781-785.)—From 2 to 3 grammes of the oxidized oil are saponified with 10 c.c. of 8 per cent. alcoholic potash on a boiling water-bath. The alcohol is evaporated, the soap dissolved in hot water, and the solution decomposed with hydrochloric acid in a separatory funnel, shaken with 25 c.c. of petroleum spirit, and allowed to stand overnight. As the non-volatile acids are all contained in the petroleum layer, it is unnecessary to shake out again the aqueous layer with petroleum spirit. After running off the lower liquid the petroleum layer is withdrawn, leaving the oxy-fatty acids in the funnel. If their quantity is considerable, they may enclose unoxidized fatty acids, and it is therefore advisable to dissolve them in a dilute solution of soda or ammonia, and repeat the treatment with petroleum spirit after acidifying with hydrochloric acid.

The united petroleum spirit extracts are evaporated, and the residue, consisting of the unoxidized fatty acids and unsaponifiable matter, dried to constant weight (1). It is then dissolved in 25 c.c. of 90 per cent. alcohol, and titrated with seminormal alkali, the milligrammes of KOH being calculated on the original oil. The number thus obtained, which the author terms the "inner saponification value," furnishes a measure of the non-volatile and unoxidized fatty acids.

The neutral alcoholic solution is extracted with petroleum spirit, the extracts washed with alcohol, the petroleum spirit evaporated, and the residue of unsaponifiable matter dried and weighed (2).

The difference between (1) and (2) gives the quantity of non-volatile fatty acids, whose molecular weight can be calculated from the inner saponification value.

The oxy-fatty acids left in the separatory funnel are dissolved in hot alcohol, the solvent evaporated, the residue dried to constant weight, ignited, the ash deducted, and the difference taken as the oxy-fatty acids (3).

The sum of 1+3 gives the Hehner value.

The following results were thus obtained with cottonseed-oil and three oxidation products, which were prepared by exposing the oil on wash-leather for eight and twelve days respectively. The leather was cut into fragments, and extracted with cold petroleum spirit, furnishing products A and B. The second leather still contained a considerable amount of products insoluble in petroleum spirit, which were subsequently extracted from it with cold ether (B_1). This was a thick yellow oil, soluble in alcohol.

	Cottonseed.	A.	B.	B_1.
Iodine value	108·8	55·4	46·3	29·1
Acid value	2·2	13·3	13·8	33·4
Saponification value	190·4	223·1	227·5	271·3
Inner saponification value ...	186·9	128·8	128·9	74·4
Hehner value	94·22	85·34	83·62	74·20
Unsaponifiable matter, per cent.	1.10	1·11	1·28	0·72
Oxy-fatty acids, per cent. ...	0·27	20·70	19·43	37·72
Non-volatile fatty, acids per cent.	92·85	62·53	62·91	35·76
Molecular weight of fatty acids	278·1	276·2	273·2	269·1
Melting-point of fatty acids ...	35-36°	45-46°	46°	51°

With regard to these results, the author points out that the fact that volatile acids are produced during the oxidation process is shown by the decrease in the Hehner and inner saponification values. The increase in the amount of unsaponifiable matter in B is only apparent since B and B_1 are both fractions of the same oxidation products, and the greater proportion of unsaponifiable matter was removed by the preliminary treatment with petroleum spirit which gave B.

The general conclusion arrived at on this point is that during the oxidation of fats and oils the unsaponifiable matter remains intact, and new substances are not formed from it.

Unlike the oxy-fatty acids of liver-oils (*Zeit. angew. Chem.*, 1891, 643), those of cottonseed-oil are completely soluble in ether.

The foregoing method of analysis affords a means of examining the course of oxidation during the drying of linseed-oil, and is also applicable to the examination of unoxidized fats and oils, as is seen in the following examples:

	Ox-tallow.	Olive-oil.	Butter-fat.
Saponification value ...	193·9	188·4	225·9
Inner saponification value ...	193·8	188·1	185·2
Hehner value	95·58	95·25	87·60
Unsaponifiable matter ...	0·11	0·98	0·24
Oxy-fatty acids	0·13	0·18	0·14
Non-volatile fatty acids ...	95·34	94·07	87·22
Molecular weight of fatty acids	275·0	280·1	263·7

From these results, it is manifest that when, as in the case of tallow and olive-oil, the total saponification and inner saponification values are nearly identical, the amount of volatile or of oxy-fatty acids must be insignificant.

Butter-fat, on the other hand, by reason of its volatile acids, shows a considerable difference (40·7) between the two values, and the Reichert-Meissl value (36·3 for

5 grammes) can be calculated from this difference. This calculated value is higher than the normal, owing to the fact that the Reichert value only represents a portion of the total volatile acids.

In the case of wool-fat, a modification of this method is said to have given very satisfactory results, with regard to which the author promises a future communication.

C. A. M.

INORGANIC ANALYSIS.

The Accuracy of Gold Assay. J. Loevy. (*Chem. Zeit.*, 1898, xxii., 609.)—It was recently stated before the Chemical and Metallurgical Society of South Africa that the fusion and cupellation method of determining gold is only accurate provided the whole of the metal exists in the sample in the free state; but that if some of the gold is present as a soluble salt, the results are 30 or 40 per cent. too low. To investigate this point, infusorial earth was moistened with about 0·2 per cent. of gold chloride or the double chloride of gold and sodium, dried at 110° C., mixed with the flux (sodium carbonate, litharge, borax, and Rochelle salt), and submitted to the combined fusion and cupellation process in the usual manner. Employing about 0·1 gramme of gold, the figures varied from 95·8 to 98·3 per cent. of the theoretical yield; and the author considers the deficit to be due simply to the fineness of the kieselguhr, which caused loss of gold by formation of dust. He believes that the process always gives 98 per cent., more frequently 99 or 100 per cent. of the gold actually present; and as the test can be carried out on 300 or 500 grammes of material, it cannot be advantageously replaced by wet analysis.

F. H. L.

Detection and Estimation of Calcium Hydroxide in Set Portland Cement. N. N. Ljamin. (*Zap. Imp. Russk. Techn. Obschtsch.*, 1898, xxxii., 75; through *Chem. Zeit. Rep.*, 1898, 178.)—In thin sections of set Portland cement examined by transmitted light, glittering crystals are often to be seen, which appear dark under the polariscope with crossed Nicols, and which, dissolving in hydrochloric acid without effervescence, and also in 10 per cent. sugar solution, must be either calcium hydroxide or a strongly basic silicate. The author has succeeded in isolating some of this material from cement seven years old; it formed hexagonal and rhombic crystals, and agreed goniometrically with crystals of slaked lime. On analysis it gave H_2O, 25·4 per cent.; CaO, 73·4 per cent.; insoluble in HCl, 0·5 per cent.; its specific gravity was 2·18 to 2·20, or a little higher than the normal (2·07 to 2·08).

To estimate the calcium hydrate, the set cement is ground until its particles are smaller than these crystals. It is then passed through a sieve into a Hauenschild's apparatus, and treated with a mixture of benzene and methylene iodide having the specific gravity 3·34; the vessel being placed under the receiver of an air-pump to remove air-bubbles, which would prevent the heavier portions from sinking. The calcium hydrate that rises to the surface is collected on a filter, further quantities of the same liquid mixture are added to the residual cement till all the floating matter is recovered; and finally the lime is washed with benzene and ether, dried

at 120° C., brushed off the paper into a tared watch-glass, and weighed. As the product may be contaminated with fragments of coke, etc., it can be treated again in the Hauenschild apparatus with a mixture of benzene and methylene iodide having a specific gravity lying between 1·0 and 2·0; but the amount of carbon rarely exceeds 1 per cent. of the total mass. The lime usually contains a little more (25 or 26 per cent.) volatile matter than corresponds to the formula CaH_2O_2; and about 1·5 per cent. of alumina is present, but no silica; in fact, the foreign ingredients only cause the yield of calcium hydrate to be roughly 0·5 per cent. too high.

Another method of arriving at the same result depends on the fact that calcium hydroxide only loses its water of constitution at 450° or 480° C., whereas the other substances in set Portland cement part with their water of hydration at 160°. One sample is heated for two or three hours at 180° or 200°; in another the carbon dioxide is determined; and in a third the total volatile matter. Deducting the CO_2 and the loss on heating to 200° from the latter, the amount of water in the calcium hydroxide is left; and by employing the coefficient $\frac{CaH_2O_2}{H_2O} = 4{\cdot}1$, the proportion of free lime is given. The two processes yield closely concordant figures. F. H. L.

The Determination of Potash without the previous removal of Iron, Calcium, etc. C. C. Moore. (*Jour. Amer. Chem. Soc.*, 1898, xx., 340-343.)—This is based on the fact that it is possible to dissolve ferric, aluminic, and other chlorides in acidulated alcohol after previous conversion of the potassium into the ordinary compound with platinic chloride.

The substance is dissolved by the usual digestion with acid, the excess of the latter removed, and the residue and insoluble matter filtered off. Platinic chloride solution is added to the filtrate, which is then evaporated nearly to dryness on the water-bath as in the usual determination. From 15 to 20 c.c. of the acidulated alcohol are added, and the whole allowed to stand for two or three minutes with occasional stirring. The crystalline platinum potassium salt, and part of the neutral sulphates, sodium chloride, etc., remain undissolved, while the excess of platinic chloride, and the chlorides of iron, aluminium, etc., pass into solution. The insoluble residue is washed, first, with the acidulated alcohol, and then with the Lindo ammonium chloride solution, until the sulphates, sodium chloride, etc., are washed out. The ammonium chloride is then removed by washing with 85 per cent. alcohol, and the pure potassium chlorplatinate salt left on the filter is dried and weighed as usual.

The acidulated alcohol is prepared by passing dry hydrochloric acid gas into cool 90 per cent. alcohol until 1 c.c. of the latter neutralizes about 2½ c.c. of normal potash. Some ethyl chloride is formed, but most of the gas remains in solution in an uncombined state. The solubility of potassium platinic chloride in this liquid is about 1 in 60,000 at 80° C. The results obtained by this method in experiments on the ash sample used by the Association of Official Agricultural Chemists in 1897 varied on the average 0·01 per cent. from the result found by the association.

C. A. M.

Colorimetric Estimation of the Density of Chimney Smoke. P. Fritzsche. (*Zeit. anal. Chem.*, 1898, xxxvii., 92-94.)—A small glass filter tube similar to those used in gravimetric determinations of sugar is loosely packed with 2 grammes of cellulose, and is connected by means of indiarubber tubing with another glass tube sufficiently long to project a little way into the interior of the chimney when introduced through a hole in the wall. By means of an aspirator attached to the other end of the filter-tube, from 10 to 20 litres of the gases are drawn through the cellulose. The tube is then wiped out with a clean portion of the cellulose, and the whole of the latter shaken in a stoppered flask with 200 c.c. of water, until it becomes a uniformly coloured grey pulp. This is transferred to a round-bottomed test-tube (40 to 50 mm. in diameter), and the amount of soot estimated by comparing the colour with those of a standard colour scale.

This colour scale is prepared by shaking 2 grammes of cellulose mixed with different proportions of soot (from 5 to 30 milligrammes) in 200 c.c. of water, and comparing the colour of the pulp in the manner described above with that of discs tinted to various shades with Indian ink. Once standardized, the discs are pasted on to cardboard, and a note made of the amount of soot to which they correspond.

C. A. M.

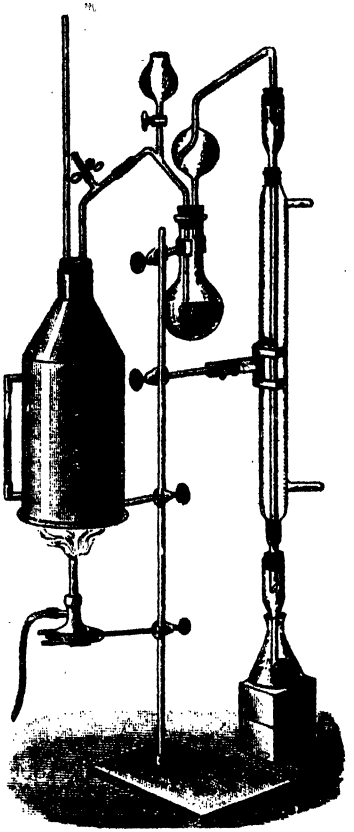
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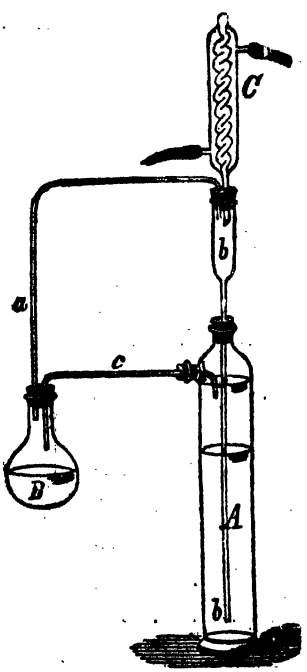
Pipette with Automatic Adjustment. H. Bremer. (*Zeit. für Untersuch. der Nahr. und Genussmittel*, 1898, 318.)—The pipette consists of two parts—the measuring tube, or pipette proper, and the stopcock attachment. The measuring tube has the usual two tubulures, which are of equal dimensions, and terminate in very small orifices; it is so constructed that it shall hold exactly the required quantity of fluid when quite full from end to end; by means of a caoutchouc stopper it is attached to the other part of the apparatus, as shown in the figure. To fill the pipette, the point is dipped into the fluid, the stopcock opened, and the fluid sucked up until it overflows at the upper orifice of the measuring tube, when the stopcock is shut off. The liquid will, of course, continue to overflow until equilibrium is re-established between the inner and the outer pressure. The pipette is discharged by turning on the stopcock. Fluids can be measured much more exactly by this pipette than by the ordinary form, since the measurement is quite independent of errors of reading off. The pipette can be made of any size or to suit any specific purpose. For measuring corrosive liquids, it is recommended that the apparatus be blown in one piece.



H. H. B. S.

New Apparatus for the Determination of Nitrogen by Kjeldahl's Method. H. Bremer. (*Zeit. für Untersuch. der Nahr. und Genussmittel*, 1898, 316.)—In this apparatus, the same flask is used for the digestion in hot sulphuric acid as for the distillation, thus obviating the necessity of transferring from one vessel to another. The flask is of about 250 c.c. capacity, and has a long neck 35 mm. wide. The digestion in this flask is said to occupy scarcely any longer time than in the flask usually employed for the purpose. As soon as the digestion is completed, the flask is cooled and 100 c.c. of water added cautiously, a little at a time, to prevent boiling. The flask is then again cooled and attached to the rest of the apparatus as shown in the figure. The soda solution is introduced through the funnel and the distillation carried out by means of a current of steam. The apparatus can also be used for the determination of ammonia by distillation with magnesia or barium carbonate. H. H. B. S.





Apparatus for the Extraction of Liquids with Immiscible Solvents. O. Foerster. (*Chem. Zeit.*, 1898, xxi., 421.)—This apparatus, which is sufficiently explained by the accomapnying diagram, is primarily intended for the extraction of unsaponified matter from soap solutions, and it is capable of recovering 99·97 per cent. of a neutral substance (cholesterin) present in a stearin soap within a period of four hours. A small quantity of aqueous liquid gradually collects in B, which must be removed at intervals, while the ethereal solution finally requires four or five washings with water. The syphon *c* should not extend into the liquid in B.

F. H. L.

APPOINTMENT.

Mr. W. Lincolne Sutton has been appointed Public Analyst for East and West Suffolk, Ipswich, and Bury St. Edmunds, in succession to the late Mr. James Napier.

THE ANALYST.

NOVEMBER, 1898.

PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS.

NOTE ON THE EXAMINATION OF LINIMENT OF CAMPHOR.

By Norman Leonard, B.Sc., F.I.C., and H. Metcalfe Smith, F.I.C.

Liniment of Camphor has long been commonly known as "Camphorated Oil," but this synonym has only been made official in the *British Pharmacopœia* of 1898. The latter, as also the editions of 1867 and 1885, directs 1 ounce of camphor to be dissolved in 4 fluid ounces of olive oil, but no characters or tests for the finished preparation are given. Taking the specific gravity of olive oil as 0·9165, this being the mean of the limits (0·914—0·919) allowed for this oil by the *Pharmacopœia*, it is easy to calculate that the liniment should contain 21·45 per cent. by weight of camphor. We have recently met with several specimens which were deficient in camphor, one sample containing only 3 per cent. of this substance, and in two cases a mineral oil was found to have been substituted for olive oil.

The amount of camphor present is most simply and accurately found by determining the loss in weight experienced by the sample on heating. For this purpose 3 to 5 grammes of the camphorated oil are heated for two hours at 120° C. in a flat-bottomed dish or flask. Olive oil under the same conditions suffers a *gain* in weight of 0·15 per cent., and hence this figure should be *added* to the loss in weight on heating the sample, in order to obtain the true amount of camphor present. The results obtained by this method are very satisfactory, as will be seen from the following table, in which are given analyses of four solutions prepared by dissolving known weights of camphor in olive oil of 0·9164 specific gravity:—

	Camphor taken, Grammes.	Olive Oil taken, Grammes.	Camphor calculated, per Cent.	Loss on Heating, per Cent.	Corrected Loss = Camphor, per Cent.	Specific Gravity at 60° F. (H_2O at 60° = 1).
1 ...	2·361	75·918	3·01	2·93	3·08	0·9178
2 ...	5·346	70·578	7·04	6·78	6·93	0·9195
3 ...	14·530	69·912	17·21	17·10	17·25	0·9241
4 ...	15·856	54·146	22·65	22·38	22·53	0·9266

An approximation to the amount of camphor may be made by determining the specific gravity of the sample, each per cent. of camphor, as may be seen from the results given above, raising the specific gravity by about 0·00045. The following formula may be used, it being remembered that the results obtained will be of little

value if oil having a specific gravity much differing from 0·9165 has been used in the preparation of the sample :—

$$\text{Camphor per cent.} = \frac{\text{specific gravity of sample} - 0{\cdot}9165}{0{\cdot}00045}.$$

Below are given the figures obtained from four specimens of "Liniment of Camphor," No. 1 being prepared by ourselves, and the remaining three procured from chemists and druggists. As already observed, the directions given by the *Pharmacopœia* correspond to the presence of 21·45 per cent. of camphor :

	Specific Gravity at 60°.	Camphor from Loss on Heating (corrected).	Camphor calculated from Specific Gravity.
1 ...	0·9264	21·35	22·2
2 ...	0·9255	20·65	20·0
3 ...	0·9257	20·46	20·4
4 ...	0·9264	20·37	22·2

As regards the presence of mineral oil, this is usually indicated by the marked bluish fluorescence of the sample. It may be further identified, and separated from any vegetable oil which may also be present, by heating for some time with alcoholic potash, the liquid being afterwards diluted with water and filtered. The mineral oil remaining on the filter is washed with water, dried, and transferred to a weighed flask by means of a little ether, which is subsequently removed by evaporation.

We are indebted to Dr. Thomas Stevenson for permission to make use of results obtained in his laboratory.

THE RELATION BETWEEN THE SPECIFIC GRAVITY AND THE INSOLUBLE FATTY ACIDS OF BUTTER AND OTHER FATS.

By Norman Leonard, B.Sc., F.I.C.

The existence of a general correspondence between the specific gravity and the percentage of insoluble fatty acids yielded by butter and margarine is well known, a low specific gravity being, as a rule, associated with a high percentage of insoluble fatty acids, and *vice versâ*. Occasionally, abnormal results are obtained, more especially the conjunction of a high percentage of fatty acids with a comparatively high gravity. It is obvious that no natural or necessary connection exists between the two analytical data, since the fats used to adulterate butter vary in character, and the rough correspondence actually observed is due to the fact that the usual adulterants (beef and mutton fat, etc.) have approximately equal densities and yield the same proportion of insoluble fatty acids. The presence of a vegetable oil, such as cotton-seed, cocoanut, or sesamé oil, having a higher density than animal fats, but yielding nearly the same proportion of fatty acids, would be indicated by a departure from the usual relation. The investigation of the latter may, therefore, be of some value to the analyst.

Four years ago I found, on examination of the analyses of thirty-three samples of butter, that the results might be very well represented by the formula $y = k(1 - x)$, where y is the percentage of insoluble fatty acids, x the specific gravity at 100° F.

(water at 60° F. = 1), and k a constant, the mean value of which was found to be 951±1·6. I have recently examined the results obtained from thirty samples analysed during the year 1897, and found k to have the mean value 951±1·8. This close agreement with the previous result is interesting as showing the constancy of the general character of the adulterants employed. The following table is calculated from the formula $y = 951(1 - x)$:

Specific Gravity at 100° F. (H_2O at 60° F. = 1).	Insoluble Fatty Acids.	Specific Gravity at 100° F. (H_2O at 60° F. = 1).	Insoluble Fatty Acids.
·8990	96·1	·9040	91·3
·8995	95·6	·9045	90·8
·9000	95·1	·9050	90·3
·9005	94·6	·9055	89·9
·9010	94·1	·9060	89·4
·9015	93·7	·9065	88·9
·9020	93·2	·9070	88·4
·9025	92·7	·9075	88·0
·9030	92·2	·9080	87·5
·9035	91·8	·9085	87·0

Of the sixty-three samples examined, the percentage of insoluble fatty acids found by experiment differed from that calculated from the formula,

By not more than 0·5 per cent. in thirty-five cases, or 56 per cent.

By not more than 1·0 per cent. in fifty-four cases, or 86 per cent.

There were only three cases in which a difference of more than 1·4 per cent. was observed, and in each of these the insoluble fatty acids were abnormally high.

The results discussed above were obtained in the laboratory of Dr. Thomas Stevenson, to whom I am indebted for permission to make use of them.

ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

FOODS AND DRUGS ANALYSIS.

Detection of Cotton-Seed Oil in Lard by Means of Phytosterin. A. Bömer. (*Zeit. der Untersuch. der Nahr. und Genussmittel*, 1898, 544.)—In two previous papers (*Zeit. für Untersuch. der Nahr. und Genussmittel*, 1898, 21, *Analyst*, xxiii., 42; *Zeit. für Untersuch. der Nahr. und Genussmittel*, 1898, 81, *Analyst*, xxiii., 132) the author published a method for the extraction of phytosterin and cholesterin, and showed that the phytosterin of vegetable fats, particularly that of cotton-seed oil, possesses sufficient characteristic differences from the cholesterin of animal fats to enable small quantities of the former to be detected in the presence of the latter. He now applies this principle to the detection of cotton-seed oil in lard.

The proof of the presence of phytosterin in cholesterin rests upon the melting-point and the crystalline structure of each body. The crystalline structure is, however, of itself sufficient evidence, should the quantity of phytosterin be too small to decidedly affect the melting-point of the mixture. The crystals of such mixtures

consist of short, apparently rounded needles, in part attached to one another in telescope fashion. Experiments made by slow crystallization upon larger quantities, however, yielded crystals in the form of three-sided prisms with flat ends. The results of the author's study of the melting-points of the two bodies have already been published (*cf. Analyst*, xxiii., 132). He now gives the results of a large number of observations of the melting-points and crystalline form of various mixtures of lard and cotton-seed-oil, which show that admixtures of 3 to 4 per cent., and, under favourable conditions, even 1 to 2 per cent., of cotton-seed oil in lard can be detected in this way.

The author claims that this mode of detection is easier and quicker of execution than the determination of the iodine number of the liquid fatty acids.

H. H. B. S.

Estimation of Glycogen in Meat Extracts. Lebbin. (*Pharm. Zeit.*, 1898, xliii., 519; through *Chem. Zeit. Rep.*, 1898, 214.)—Twenty-five grammes of the sample are dissolved in 100 c.c. of water, mixed with 100 or 150 c.c. of a 4 per cent. solution of caustic potash in 90 per cent. alcohol, and allowed to rest for one or two hours. The precipitate is filtered off, washed with 90 per cent. spirit, dissolved off the paper with 50 c.c. of water, and the solution faintly acidified with weak hydrochloric acid. It is next precipitated with about 10 c.c. of a solution of potassium mercuric iodide, filtered after half an hour, and the insoluble matter washed with hot water. From the filtrate the glycogen is thrown down on the addition of an equal volume of 95 per cent. alcohol, collected on a tared filter, washed with alcohol and ether, dried and weighed. The double mercuric iodide is prepared by dissolving 20 grammes of mercuric chloride in 300 c.c. of water, mixing with 20 grammes of potassium iodide also dissolved in 100 c.c., and adding more mercuric chloride as long as the precipitate continues to disappear.

F. H. L.

ORGANIC ANALYSIS.

The Characteristics of Olive-Kernel Oil. O. Klein. (*Zeit. angew. Chem.*, 1898, 847-850.)—According to Benedikt and other authorities, this oil differs from olive oil in having a sharp and bitter taste and a dark green colour, and in being readily soluble in glacial acetic acid and alcohol on account of the large percentage of free acids which it contains.

The author finds that this curious difference between the oils extracted from different parts of the same fruit is largely to be attributed to the manner in which the oil is obtained. After crushing the fruit so as to leave the stones intact, the mass is subjected to more or less complete expression in bags of esparto grass. The residual mass (*bagasses*) is left aside for a considerable time, often weeks, and is subsequently ground again and expressed or extracted. Under these circumstances, it is not surprising that considerable oxidation takes place in the moist mass, and that the oil frequently contains as much as 50 to 60 per cent. of free fatty acid, and is of a dark colour from the oxidation of the chlorophyll of the fruit.

A specimen of "kernel" oil thus obtained gave the following results on analysis:

Specific gravity, 0·9277; iodine value, 71·57; saponification value, 190·5; free fatty acid, 71·12 per cent.

The author prepared specimens of the pure kernel oil by hot and cold expression, and obtained the following analytical results with them: Specific gravity, 0·9186 to 0·9191; iodine value, 86·99 to 87·78; saponification value, 182·3 to 183·8; free acids, 1·00 to 1·78; refractive index, 1·4682 to 1·4688.

From these results it appears that the specific gravity and iodine value of pure olive-kernel oil are somewhat higher than is usually the case with olive oil. Only in exceptional instances and with oil obtained from such varieties as Seville, Cordova, and Manzanilla olives, is so high an iodine value given by olive oil. The saponification value is rather lower and the percentage of free acid higher than in olive oil. The percentage of free acids in the oil obtained from sound fresh fruit varied from 0·4 to 0·8 per cent., and the author attributed the difference to oxidation during the time which elapsed between the removal of the endocarp and expression of the oil.

In order to determine whether the presence of kernel oil in olive oil caused any depreciation in the latter, experimental mixtures were prepared in 1891 and 1892, and preserved in well-stoppered bottles in the dark until 1898. The samples showed scarcely any change in colour, taste, or smell, and the amount of free acids had only increased by about 0·2 per cent.

The author examined a specimen of olive-kernel oil by Hazura's method, and found it to contain 9·7 per cent. of solid fatty acids (consisting of 40 per cent. of stearic acid and 60 per cent. of palmitic acid). The liquid fatty acids were found to consist of oleic and linolic acids. Arachidic acid was not detected in the solid fatty acids.

The general conclusions arrived at from the research were:

1. The characteristics hitherto assigned to olive-kernel oil are really those of *bagasses* oil.

2. The physical and chemical characters of pure olive-kernel oil are very similar to those of olive oil.

3. Olive-kernel oil is as stable as olive oil, if proper precautions are taken.

4. It can be mixed with olive oil without ill effects, and there is no reason why the stone and the fruit should not be crushed and pressed together. C. A. M.

Separation of Unsaturated from Saturated Fatty Acids. K. Farnsteiner. (*Zeit. für Untersuch. der Nahr. und Genussmittel*, 1898, 390.)—The author's method is founded upon the fact that warm benzene dissolves the lead salts of the solid as well as the liquid fatty acids, but that on cooling, the salts of the solid fatty acids crystallize out almost completely, leaving the salts of the liquid fatty acids in solution. 0·6 to 1 gramme of fat is saponified with alcoholic alkali in an Erlenmeyer flask, the solution neutralized with acetic acid, using phenolphthalein as indicator, the alcohol almost completely expelled by evaporation, the soap dissolved in about 100 c.c. of boiling water, and precipitated with about 30 c.c. of a boiling solution of lead acetate containing about 1 gramme of the salt. By agitating the flask in cold water in the usual way, most of the lead salts are separated from the fluid in a compact mass.

After complete cooling, the filtrate is removed by decantation through a moistened filter, leaving the bulk of the lead salts in the flask. This, as well as the small quantity on the filter, is then washed with cold water, after which that upon the filter is washed back into the flask. The flask is now placed upon a bath of boiling water, when in a short time the lead salts will be found to melt together into a compact mass. By agitating the flask as it cools, any remaining crystals of the lead salts can be made to adhere to the sides of the flask, and the liquid can then be poured off clear. The salts are now as far as possible dried by means of a roll of filter-paper, and are then dissolved in 50 c.c. of moderately warm benzene. The solution is allowed to stand for about fifteen minutes at the temperature of the laboratory, in order to obtain a coarse crystalline separation, and is then kept for two hours at 8° to 12° C.

The filtration is conducted in the following manner: The flask is closed by a tight-fitting cork, through which two glass tubes pass, one a straight piece about 10 cm. long, reaching about 1 cm. into the flask, and the other bent in the form of a syphon. Into the open end of the bent tube inside the flask is fixed a moderately thick wad of cotton-wool, free from fat, extending a few millimetres outside the orifice. The short vertical tube is connected with a caoutchouc ball. By compressing the air within the flask by means of the ball, the liquid is forced through the plug of cotton-wool up the bent tube, and can be collected in a flask placed to receive it. When the liquid has been thus removed, the cork is lifted, and about 10 c.c. of benzene, at a temperature of about 10° C., is run into the flask. This is agitated with the precipitate, and then removed by compressing the caoutchouc ball as before. Twenty-five c.c. of benzene are then added, the bent tube lifted out of the liquid, the benzene heated to boiling to dissolve the precipitate, then cooled for one hour at 8° to 12° C., and removed by filtration through the plug as before. This operation is repeated yet again with 25 c.c. of benzene, altogether from 120 to 130 c.c. of benzene filtrate and washings being obtained.

For the separation of the liquid fatty acids, the filtrates are shaken in the usual way with an equal volume of 10 per cent. hydrochloric acid, until the salts are completely decomposed, and after two washes with water, the solution of the fatty acids is filtered through wadding and distilled in a current of hydrogen.

For the separation of the solid fatty acids, 25 to 30 c.c. of benzene are added to the lead salts in the flask, the bent tube lifted above the level of the fluid, and the benzene heated for a short time to the boiling-point. The flask is then removed from the water-bath, and the pressure-tube closed with the finger, causing air to enter through the cotton-wool plug owing to the condensation of the benzene vapour. Most of the liquid in the tube and plug is thus sucked back into the flask. The plug is then withdrawn and thoroughly extracted by boiling in a test-tube several times with a few c.c. of benzene. Finally, the lead-salts are decomposed by heating the benzene solution for fifteen minutes with a 10 per cent. hydrochloric acid solution, and the same treatment pursued as in the case of the liquid fatty acids, except that the use of hydrogen in the distillation of the benzene is omitted.

In the case of the separation of free fatty acids, the author converts them into the lead salts by means of hydrated lead oxide, prepared by precipitating lead acetate

with sodium hydrate, washing with water, alcohol and ether, drying at a gentle heat, and reducing to a fine powder. For 1 part by weight of the solid fatty acids, about 0·4 part of the hydrated lead oxide are required, and for 1 part of the liquid fatty acids about 0·2 part. An excess of the hydrated lead oxide is without influence upon the results.

The following results show the degree of accuracy obtainable :

No.	Quantity in Grammes taken for the Analysis.	C.c. of Benzene used.	Number of Precipitations.	Liquid Acids.		Solid Acids.		Remarks.
				Taken, per cent.	Found, per cent.	Taken, per cent.	Found, per cent.	
1.	0·7260	120	4	19·1	17·45	80·9	82·4	Old oleic acid.
2.	0·6646	120	4	21·35	22·4	78·65	77·5	Ditto.
3.	0·9155	100	4	33·75	30·8	66·25	67·9	Ditto, first filtrate allowed to stand overnight.
4.	0·9104	150	3	44·0	42·5	56·0	—	Ditto.
5.	0·6456	100	3	47·1	45·05	52·9	54·7	Liquid acid from olive oil.
6.	0·6190	90	3	51·2	48·8	48·8	48·7	Ditto, first filtrate allowed to stand overnight.
	0·4960	100	3	56·8	55·8	43·2	43·3	Old oleic acid.
	0·6333	125	3	59·5	—	40·5	39·4	Liquid acid from cotton oil.
9.	0·5799	110	3	60·6	61·0	39·4	39·4	Freshly prepared, almost pure oleic acid.
10.	0·7834	90	2	91·0	89·4	9·0	10·8	

The author then developes the following argument, that the percentage of unsaturated fatty acids in a mixture of fatty acids or in a fat can also be ascertained by calculation from the iodine number of the mixture and the iodine number of the liquid portion of the fatty acids, without it being necessary, as stated by Twitchell (ANALYST, 1895, 165) to determine approximately the quantity of the latter.

Let J be the iodine number of the substance, and J_1 the iodine number of the liquid portion of the fatty acids.

Then, since J_1 parts by weight of iodine correspond to 100 parts by weight of the liquid fatty acids, it follows that 1 part by weight of iodine corresponds to $\frac{100}{J_1}$ parts by weight of the liquid fatty acids. The quantity of iodine (J) absorbed by 100 parts by weight of substance consequently corresponds to $\frac{100J}{J_1}$ parts by weight of the liquid fatty acids. The formula $\frac{100J}{J_1}$ therefore gives the percentage of liquid fatty acids in the substance.

The iodine number of a sample of lard, for example, is 54, and the iodine

number of the liquid portion of the fatty acids is 90. The sample will contain $\frac{100 \times 54}{90} = 60$ per cent. of liquid—*i.e.*, unsaturated fatty acids.

The accuracy of this method is influenced first by the accurate determination of the two iodine numbers; secondly, by the amount of loss in iodine-absorbing power suffered by the unsaturated fatty acids through oxidation, etc., during their preparation; and thirdly, by the question as to whether, in the case of the incomplete separation of the lead salts, the unsaturated acids remaining in the insoluble lead salts are sufficient to influence the iodine number of the principal quantity.

H. H. B. S.

Use of Benzene in the Determination of the Iodine Numbers of Fats and of the Liquid Portion of Fatty Acids. K. Farnsteiner. (*Zeit. für Untersuch. der Nahr. und Genussmittel*, 1898, 529.)—In a recently published paper (*Zeit. für Untersuch. der Nahr. und Genussmittel*, 1898, 390, *Analyst*, preceding Abstract) the author proposes a method for the separation of unsaturated from saturated fatty acids by means of benzene. In the present communication he shows that the iodine numbers of fats and of liquid fatty acids may be determined in the presence of benzene, if the latter be free from thiophen. Ordinary benzene, owing to the presence of this impurity, absorbs an appreciable quantity of iodine, and therefore vitiates the results. The following comparative results were obtained:

Description of Fat.	Iodine Number found in Presence of		Difference by use of Benzene.
	Chloroform.	Benzene.	
1. Lard I., prepared by author ...	49·0	48·7	−0·3
2. ,, II., from local slaughter-house	49·7	49·5	−0·2
3. Cooking fat (fabricated)	76·1	75·2	−0·9
4. Tallow I.	40·2	39·7	−0·5
5. ,, II., Australian	44·3	44·0	−0·3
6. ,, III., ,, ...	44·7	44·1	−0·6
7. Goose-fat I., pure ...	69·8	69·0	−0·8
8. ,, II.,	77·7	77·9	+0·2
9. ,, III., with lard admixture ...	72·9	72·2	−0·7
10. Sesame oil	103·9	103·1	−0·8
11. Cotton-seed oil	101·1	100·8	−0·3
12. Linseed oil	178·9	177·0	−1·9

For the determination of the iodine number of the liquid fatty acids, the author takes a portion of the mixed fatty acids corresponding to about 1 gramme of the liquid fatty acids—that is to say, about 2 grammes for lards, etc., and about 1·2 to 1·5 grammes for oils. The lead salts are produced in the usual way, and then dissolved in 100 c.c. of warm benzene, the solution allowed to stand for 10 to 15 minutes, kept for 2 hours at 8° to 12° C., and filtered without subsequent washing. The liquid fatty acids are obtained in the free state by shaking the filtrate with about 100 c.c. of a 10 per cent. hydrochloric acid solution until the layer of benzene becomes

nearly clear. After washing twice with 100 c.c. of water, the benzene solution is filtered through paper or cotton-wool without washing. From the filtrate three 25 c.c. portions are taken; two of these are used for the determination of the iodine number in the usual way; the third is used to ascertain the weight of the fatty acids taken for the determination, by distilling off the benzene in a current of hydrogen. The iodine number can also be determined in the residue from the distillation.

In the following table the results by the benzene method are compared with the results by the ether method. The two experiments in which benzene only was used are intended to show that the absorptive power of the fatty acids is not affected by the distillation in hydrogen.

No.	Description of Fat.	Iodine Number of the Liquid Portion of the Fatty Acids. Separation effected by means of					Remarks.
		Ether.		Benzene.			
		1.	2.	1.	2.	After Distilling off Benzene. 3.	
I.	Lard, prepared by author	—	—	86·5	86·5	86·5	Solution of the lead salts stood over-night without hydrogen.
II.	Ditto	90·7	—	—	—	93·0	Separation by ether carried out under heat. Without hydrogen.
III.	Lard, American	—	—	96·2	96·2	96·3	Lead salts produced by boiling the benzol solution of the fatty acid with hydrated lead oxide.
IV.	Cooking-fat, composed of tallow and cotton oil	123·4	124·1	124·5	124·5	125·3	By ether; over-night, under hydrogen. By benzene; three hours exposed to air after precipitation.
	Mean ...	123·75					
	Mixture of fatty acids	133·1	132·4	132·7	132·7	131·6	Lead salts stood over-night under hydrogen.
	Mean ...	132·75					

H. H. B. S.

The Separation of Bi-basic Fatty Acids resulting from the Oxidation of Fats. L. Bouveault. (*Bull. Soc. Chim.*, 1898, xix., 562-565.)—In the course of experiments on adipic acid, the author had occasion to separate the various fatty acids in a mixture derived from the oxidation of a fat, and consisting principally of succinic, adipic, glutaric, and pimelic acids. The following is an outline of the method which he adopted:

By distilling the mixture under ordinary pressure until the temperature reached

290° C., the whole of the succinic acid was converted into succinic anhydride, which could then be separated from the other acids by continuing the distillations under a pressure of 20 mm., when it passed over at 135° C.

The residual mixture of bibasic acids (boiling at 210° to 225° C. under 20 mm. pressure) was treated with cold ether, which dissolved about one-third of the total quantity.

The insoluble portion (M.P. 135° to 140° C.) consisted chiefly of pimelic and adipic acids, and deposited the latter on crystallization from boiling water. The adipic acid thus obtained melted at 140° C., and, though insoluble in water, was found to be extremely deliquescent.

The glutaric and pimelic acids dissolved in the ether and in the mother-liquid of the aqueous crystallization were separated by being converted into calcium salts and treated with water, in which calcium pimelate is but slightly soluble in the cold and insoluble on boiling, whilst calcium glutarate is exceedingly soluble.

The author states that Arppes' statement that pimelic acid is not present in products of the oxidation of fats with nitric acid is incorrect; in fact, in such oxidations he has found all the usual acids present in varying quantity from succinic up to sebacic acid. C. A. M

The Fractional Saponification of Oils and Fats—II. R. Henriques. (*Zeit. angew. Chem.*, 1898, 697-702.)—This paper contains some supplementary notes to the author's former communication on this subject (ANALYST, this volume, 181-183). It is stated that the conclusions arrived at as to the formation of ethyl esters of the fatty acids with liberation of the glycerin during the saponification of tri-glycerides with alcoholic potash were forestalled by Bouisin in 1857, who drew the same deductions from a similar series of experiments (*Comptes Rend.*, xlv., 35; *J. prak. Chem.*, 1857, lii., 308).

Recently Geitel (*J. prak. Chem.*, 1897, 429; 1898, 113) has come to the conclusion from mathematical and physical considerations that in the saponification of all partially decomposed tri-glycerides, mono- and di-glycerides are produced. Without attempting to refute this view on theoretical grounds, the author points out that Geitel has brought forward no experimental evidence in favour of his theory, with the exception of certain results which in his (the author's) opinion lend more support to the theory of the production of ethyl esters.

Moreover, since in his former paper the author suggested the possibility of di-glycerides being present in the residue left on saponifying linseed oil with a smaller amount of alcoholic potash than was requisite to liberate all the glycerin, he has made further experiments on this point with almond oil, and finds that only unaltered tri-glycerides are left. Even on treating almond oil for eight hours with steam at 250° to 270° C., so as to cause a disruption of the molecule and distillation of about 10 per cent. of the substance, the residual portion when washed from liberated fatty acids and glycerin was found to contain only unaltered tri-glycerides.

C. A. M.

The Determination of the Lubricating Power of Oils. E. Weiss. (*Dingler's Polyt. Journ.*, 1898, cccix., 76-80.)—The viscosity of an oil is often regarded as the

most important factor in determining the suitability of an oil for lubricating purposes, but in the author's opinion it should not be regarded as the sole criterion. By means of an apparatus which he has devised it is possible to determine the consistency of an oil, and from the result to calculate the "mobility" (*Beweglichkeit*) which furnishes a means of judging which of two oils with the same viscosity is the better lubricator.

This apparatus consists essentially of a small metal disc about 10 cm. in diameter which is made to revolve in the oil by means of clockwork actuated by a weight, the number of revolutions in a given time being dependent on the consistency of the oil. The weight is chosen so that the disc makes 150 revolutions in water at 15° C. in half a minute. Usually about 1,147 grammes are required.

On charging the vessel with a thick liquid and varying the driving weight, the resistance of the liquid is proportional to the rapidity with which the disc revolves, or $P = mu$, where P = the driving force, u = the number of revolutions in thirty seconds, and m = a constant of the oil under examination. In the case of rape oil this constant was found to be 64·27. For more mobile liquids it was determined experimentally that up to a weight of 2,500 grammes the following formula was applicable, $P = a + bu^{\frac{3}{2}}$, in which $a = 43{\cdot}5$ and $b = 0{\cdot}60853$.

For liquids whose consistency lies between that of water and rape oil, the relation between the driving weight and rapidity of revolution is more complex; but here, too, the author demonstrates that there is such a constant relation, and arrives at the formula $P = A\mu v_0 (1 - \alpha)$, in which A is a constant depending on the dimensions of the consistency apparatus, v_0 = the rapidity of revolution of the oil near the disc, μ = the coefficient of friction of the liquid, and α denotes what fraction the rapidity of revolution near the surface is of the rapidity of revolution near the disc.

For Engler's viscosimeter, which the author employs, the law of Poiseuille holds good, $T = B\mu$, in which T = the time of efflux of the liquid, and B = a constant of the particular apparatus used.

Incorporating this formula into his previous equation, the author arrives at the formula,

$$Tv_0 = \frac{P.B}{A} \cdot \frac{1}{1-\alpha}.$$

If, then, in the examination of a number of oils the same viscosimeter and consistency apparatus, and in the latter the same driving weight, are invariably used, $\frac{PB}{A}$ has always the same value, and the product of the number of revolutions and of the time of efflux in the viscosimeter must be proportional to the constant $\frac{1}{1-\alpha}$, which varies with the value of α. Thus, α and Tv_0 furnish a measure of the mobility of a fluid, since the more mobile a liquid, the less the difference between the rapidity of its revolution in different parts of the vessel. Hence the product Tv_0 is termed by the author the "mobility."

If the "mobility" were solely dependent on the coefficient of internal friction, all liquids with the same viscosity would have the same "mobility," which is not the case. The "mobility" steadily decreases as the temperature rises, and when the consistency exceeds thirty revolutions fatty oils lose their oily character.

In the case of rape oil the relation between the consistency value (U) and the viscosity value (V) can be expressed with approximate accuracy by the formula

$$(V+1{\cdot}92)\,(U-0{\cdot}08)=387{\cdot}54\,;$$

and this formula was used to calculate the figures in the fourth row of the subjoined table, which gives the comparative results obtained with that oil at varying temperatures:

No. of revolutions	17	20	24	28	30	36	40	50	56
Minutes (viscosimeter)	21	17·3	14·2	12·0	11·15	9·0	7·9	5·8	4·5
Tv_0	357	346·0	340·8	336·0	334·5	324·0	316·0	290·0	252·0
Minutes (calculated)	20·98	17·53	14·28	11·96	11·03	8·87	7·78	5·84	5·01

The curves of the changes in the viscosity and consistency caused by dilution are parallel to the curves of those brought about by heat.

The following tables give in an abridged form some of the determinations made by the author in illustration of the foregoing points:

I.

Revolutions in Consistency Apparatus.	Glycerin, Specific Gravity 1·233 at 20° C.			Rape Oil.			Rape Oil and Colophony, 10 : 1.			Liquid Paraffin.		
	Minutes, Viscosity.	Temp.	Mobility.	Viscosity.	Temp.	Mobility.	Viscosity.	Temp.	Mobility.	Viscosity.	Temp.	Mobility.
		° C.			° C.			° C.			° C.	
10 ...	—	—	—	—	—	—	—	—	—	31·2	10·6	312·0
12 ...	—	—	—	—	—	—	—	—	—	26·5	13·3	318·0
14 ...	19·0	17·4	266·0	—	—	—	—	—	—	22·5	15·6	315·0
18 ...	14·65	21·4	263·7	19·7	9·0	354·6	—	—	—	16·7	19·8	300·6
24 ...	10·8	26·0	259·2	14·2	16·2	340·8	13·85	25·6	324·4	12·3	24·6	295·2
26 ...	10·0	27·0	260·0	13·0	18·2	338·0	12·85	27·6	334·1	11·4	25·8	296·4
36 ..	6·6	34·0	237·6	9·0	25·6	324·0	8·7	36·7	313·2	8·4	30·9	302·4
48 ...	4·15	43·4	199·2	6·2	35·4	297·6	6·0	46·5	288·0	6·0	37·7	288·0
56 ...	2·45	54·0	137·2	4·50	45·0	252·0	—	—	—	5·2	40·8	270·4

Revolutions in Consistency Apparatus.	Mineral Oil I.			Mineral Oil II.			Liver Oil.			Castor Oil.		
	Minutes, Viscosity.	Temp.	Mobility.	Viscosity.	Temp.	Mobility.	Viscosity.	Temp.	Mobility.	Viscosity.	Temp.	Mobility.
		° C.			° C.			° C.			° C.	
10 ...	—	—	—	—	—	—	—	—	—	—	—	—
12 ...	—	—	—	—	—	—	—	—	—	—	—	—
14 ...	20·6	26·6	288·4	22·8	51·2	319·2	—	—	—	—	—	—
18 ...	15·6	30·3	280·8	17·7	56·2	317·6	—	—	—	16·4	51·3	295·2
24 ...	12·1	34·8	290·4	13·1	62·3	314·4	12·4	7·9	297·6	12·3	57·0	295·2
26 ...	11·2	36·0	291·2	12·0	64·2	312·0	11·5	9·5	299·0	11·6	58·4	301·6
36 ...	7·6	42·8	273·8	—	—	—	7·8	17·7	280·8	—	—	—
48 ...	5·0	50·4	260·0	—	—	—	5·5	27·5	264·0	—	—	—
56 ...	—	—	—	—	—	—	4·0	37·0	224·0	—	—	—

II.

	No. of Revolutions in Consistency Apparatus.	Viscosimeter Minutes.	Mobility.
Melted lard at 41·2°	52	4·8	249·6
,, ,, 43·8°	54	4·35	234·9
,, ,, 47·2°	56	3·9	218·4
Lubricating soap dissolved in water and spirit at different temperatures :			
(*a*)	22·1	12·8	282·9
(*b*)	24·1	10·15	244·6
(*c*)	61·4	2·5	153·5
Aqueous solution of gum arabic :			
(*a*)	13·65	19·9	291·6
(*b*)	31·8	8·6	273·5
(*c*)	59·6	6·4	453·4
Soluble glass	35·5	5·6	198·8

A comparison of these results shows that as regards "mobility" the substances in the tables stand in the following ascending order: Soluble glass, glycerin, soap solutions, gum solutions, oils. The latter, compared with one another, have the following classification: Rape oil, rape oil with colophony, melted lard, mineral oil II., liquid paraffin, castor oil, liver oil, mineral oil I. A characteristic property of castor oil is that its "mobility" increases as it becomes more fluid. C. A. M.

Some Properties of Pure and Adulterated Wax. K. Dieterich. (*Chem. Zeit.*, 1898, xxii., 729.)—The author finds that the iodine number of pure yellow beeswax, as determined by the ordinary Hübl process, varies between 8·8 and 10·7; employing the Waller method, the figures are 8·4 and 10·5 respectively. As the absorption is small, it is better to use 0·5 or 0·75 gramme for analysis, dissolving it in 40 c.c. of chloroform, and allowing the mixture to rest overnight before adding the iodine solution. Yellow wax, whether filtered or not, is completely soluble in chloroform; but white wax is not entirely dissolved. The iodine number of white wax is 4·2 to 4·4 (Hübl), 4·0 to 4·2 (Waller). A wax-like residue obtained in the manufacture of wool-grease has recently appeared on the market; and as it is much cheaper than beeswax, it may possibly be used as an adulterant. Its iodine number is 7·8 to 14·2 (Hübl), 3·1 to 3·7 (Waller); chloroform does not dissolve it wholly.

A genuine sample of yellow wax was mixed with 10 and also 20 per cent. of various foreign substances of similar nature (*cf.* table), the final iodine number of the product being determined in each case. Most of the bodies selected for trial affect the absorption one way or the other; but Carnauba and Japan wax make practically no difference. The former, however, forms a mixture which is only partly soluble in chloroform; while beeswax sophisticated with Japan wax is entirely soluble. The addition of ceresin or paraffin also causes imperfect solubility. On the whole, it would seem that the iodine absorption of true wax is subject to such wide variations that by itself it cannot always be relied upon to indicate the presence of impurity; that the degree of solubility in chloroform is a most important factor; and that to

ensure even reasonable accuracy in the valuation of any particular sample, it is necessary also to deduce the acid, ester, and saponification numbers of the material. The annexed table shows the general result produced on the constants of genuine wax by the several adulterants quoted:

	Specific Gravity.	Acid No.	Ester No.	Saponification No.,	Iodine No.
Paraffin	raised	raised	raised	raised	lowered
Stearic acid ...	raised	raised	—	raised	raised
Ceresin	—	lowered	lowered	lowered	lowered
Carnauba wax ...	raised	lowered	—	—	unchanged
Japan wax ...	raised	—	raised	raised	unchanged
Lard	lowered	—	raised	raised	raised
Beef tallow ...	lowered	—	raised	raised	raised
Colophony ...	raised	raised	lowered	raised	raised

F. H. L.

Extraction of Cholesterin and Phytosterin. H. Kreis and O. Wolf. (*Chem. Zeit.*, 1898, xxii., 805.)—Although the process described by Bömer (ANALYST, xxiii., 42) affords most valuable information as to the derivation of a fat, it is lengthy, complicated, and, in hot weather, unpleasant owing to the large volumes of ether consumed. If only 10 grammes of the sample are taken for analysis, as Bömer has more recently suggested, the product is almost too small for proper examination; and Raumer's idea of evaporating the soap solution and extracting the dry residue with ether is not so easy to carry out as it sounds. The authors hope that the following modification will prove more simple and acceptable: 50 grammes of fat are saponified with alcoholic caustic soda. The solution is evaporated in a porcelain basin till it becomes syrupy, poured into 500 c.c. of boiling water in a 2-litre flask, and mixed with hydrochloric acid (sp. gr. 1·124) until phenolphthalein shows that the liquid is only just alkaline. 100 c.c. of a 10 per cent. aqueous solution of calcium chloride are then added, and the whole is shaken till the lime soaps cohere into lumps. (Absence of frothing shows that sufficient of the reagent has been employed.) It is next cooled to the ordinary temperature, filtered through cloth, and the precipitate squeezed dry in paper. Thus prepared the soaps form a friable mass and contain the whole of the cholesterin and phytosterin. The product is powdered, boiled for an hour with 100 c.c. of 95 per cent. alcohol, cooled and filtered. The filtrate is treated with 3 c.c. of 40 per cent. soda to attack any saponifiable matter left behind on the first occasion, and evaporated slowly to dryness. The residue is broken up and shaken for an hour with 50 c.c. of ether, filtered and evaporated once more. This final product is readily soluble in hot spirit; and on cooling, the cholesterin and phytosterin separate out pure and white.

Bömer has remarked (ANALYST, xxiii., 132) that the cholesterin of butter is difficult to obtain pure; the present process is available in this case also. Hitherto it has been tested on butter, lard, and cotton oil; details will follow in a subsequent communication. F. H. L.

Reaction of Lecithin with Alloxan. N. Orlow. (*Farmaz. J.*, 1898, xx., 283; through *Chem. Zeit. Rep.*, 1898, 233.)—If alcoholic solutions of lecithin and alloxan

(which must be free from nitric acid) are mixed together, the liquid turns pink, red, and dark, a voluminous red precipitate being formed. The reaction proceeds almost instantaneously on warming. The precipitate is readily soluble in water, decomposing on evaporation even in the desiccator into a brown powder resembling murexide. Ammonia does not affect the colour. The aqueous solution of the precipitate yields a violet or blue deposit with lead acetate and ammonia. Cholesterin and fats give the reaction only on heating and in a far less marked degree. Pure cholesterin does not change in colour more quickly than the alcoholic solution of alloxan alone.

F. H. L.

The Tornoe Spectro-areometric Method for Estimating Alcohol and Dry Extract in Beer. **M. Buisson.** (*Rev. Chim. Analyt. Appl.*, vol. vi. [10], pp. 157-164.)—The apparatus (Fig. 1) consists of a special refractometer fitted with a graduated circle and vernier for reading off the refraction in degrees, minutes, and seconds, and employing a Hallwachs' prism (A) illuminated by a monochromatic (sodium)

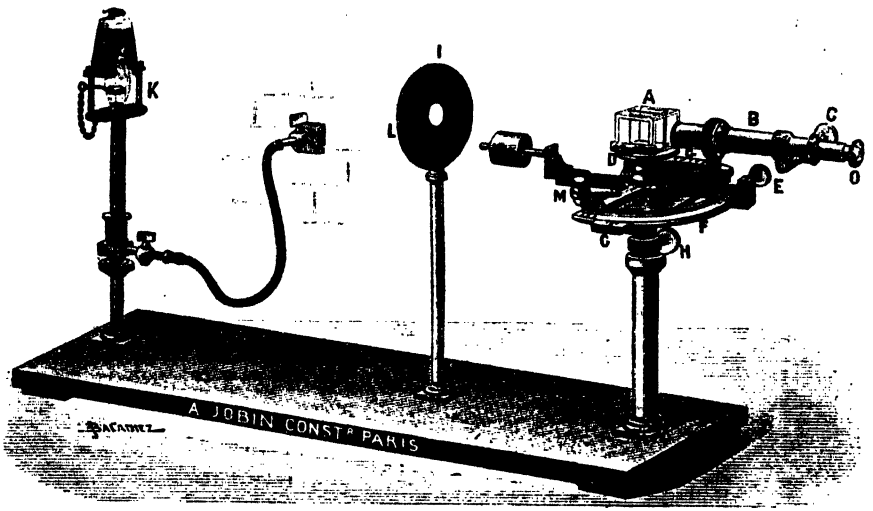
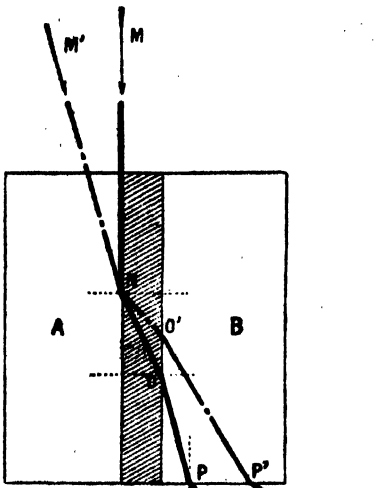


FIG. 1.

flame (K). The prism consists of a rectangular vessel divided by a partition into two compartments, one of which is filled with distilled water, and the other with the beer under examination previously freed from carbon dioxide. Equalization of temperature in the contents of the prism is ensured by agitating the whole before each reading. Of the light thrown on the prism (Fig. 2) in the direction of the partition, the rays falling on the partition are deflected by the latter and refracted through the beer, their course being indicated by M N O P R, whilst the other rays follow the direction M' N' O' P' R', the field to the right of M N being thus illuminated, whilst that to the left of the partition remains dark. The line of demarcation (M N, Fig. 3) is well defined, and its direction can be accurately determined by the aid of the cross wires in the apparatus.

In working the apparatus the prism is first carefully cleaned, filled, and put in position on the stand, the base of the eye-tube B (Fig. 1) being placed in contact with the tip of the adjusting screw E, which is then turned until the zero of the vernier coincides with the 180° mark on the scale. The eye-tube is then trained on to the lamp-flame, and the prism adjusted so that the partition is in line with the mark I on the condenser L, the part filled with water being on the left hand. The eye-piece is focussed on the cross wires, and the eye-tube—still in contact with the screw E—is turned so as to bring its extremity opposite to the compartment containing the beer, the scale also being rotated at the same time. This procedure brings into the field of vision the line of demarcation, M N (Fig. 3), the ill-defined section at the extreme right being, if necessary, placed out of view by simply turning the scale and attached apparatus in the proper direction. The platform carrying the prism is rotated so as to bring the line M N in line with the intersection of the cross wires, whereupon the position of the prism in relation to the 180° mark on the scale is noted. The prism is then rotated 180°, and the position of the line M N—now to the left of the central partition—is ascertained as before, the angle of deviation indicated by the position of the zero mark of the vernier being then read off and referred to the table which gives the corresponding quantity of dry extract.

Fig. 2.



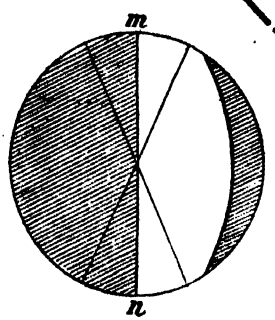


Fig. 3.

The standard temperature for the operation being fixed at 17·5° C., the following corrections must be made for other temperatures, viz., for deviations of 15°, 20° and 30° an addition of 0·7, 1·0, and 1·2 respectively for each degree C. above 17·5° C., and a corresponding deduction in cases where the temperature is below that standard, as determined, at the moment of reading off, by a thermometer inserted in the contents of the prism.

Specific gravity is estimated by the usual instrument at 17·5° C., with a correction of ±0·0002 for each degree C. above or below that limit.

Tables of estimations made with the Tornoë instrument and by the direct method, by Fernbach, Kjeldahl, and Prior, show that the differences between the two fall within the limits of experimental error. C. S.

Sugar Estimation by the Kjeldahl Process. Bruhns. (*Centralbl. Zuckerind.*, 1898, vi., 817 and 895; through *Chem. Zeit. Rep.*, 1898, 229.)—When Kjeldahl's method of estimating sugar is adopted for mixtures containing cane sugar, the copper thrown out of solution by the cane sugar is so much more copious that, other things being equal, 200 milligrammes of metal are only about equivalent to 44 milligrammes in the Soxhlet process. This depends on the longer boiling and the greater alkalinity of the modified Fehling's solution—two factors which Kjeldahl has completely overlooked. It therefore leads to quite erroneous results in the analysis of raw sugar and articles of food, etc.; and before it could be made serviceable, would require thorough investigation. It is certainly precluded from ranking as an official method. (See also ANALYST, xxiii., 271.) F. H. L.

Determination of Methoxyl. G. Gregor. (*Monatshefte für Chemie*, xix., 116.)—The author introduces sundry modifications in Zeisel's method for the determination of methoxyl (*Monatshefte für Chemie*, 1885, 989; ANALYST, xi., 119), with the object of rendering it easier and quicker of execution and more suited to technical purposes. In the first place he abolishes the direct gravimetric determination of the silver iodide, substituting for it the equally exact but quicker volumetric method of Volhard. Next he replaces the neutral silver nitrate solution of Zeisel's original method by a solution acidified with nitric acid. As Zeisel himself observed, not only does the precipitated silver iodide carry down a certain quantity of silver nitrate, but the silver nitrate solution contains traces of silver iodide. As a consequence, the precipitate and solution, according to Zeisel's procedure, require to be separately treated, the former by agitation with water to extract the silver nitrate, and the latter by evaporation to expel the alcohol, after diluting with water and acidifying with dilute nitric acid. By the use of a silver nitrate solution acidified with nitric acid the necessity for such separate treatment is avoided, for although the silver iodide carries down with it a little silver nitrate, this is easily washed out with water.

Lastly, the author prevents the reduction of silver nitrate which takes place in the tube, which dips into the alcoholic silver nitrate solution of the first flask, by employing a solution of potassium carbonate and arsenious acid instead of amorphous phosphorus. To suit the volumetric method of Volhard, the author uses an alcoholic $\frac{N}{10}$ silver nitrate solution standardized with a $\frac{N}{10}$ potassium thiocyanate solution. For the determination, 50 c.c. of this solution (acidified with a few drops of nitric acid free from nitrous) are placed in the first flask and 25 c.c. in the second. When the reaction is complete, the clear fluid is decanted from the silver iodide into a 250 c.c. measuring-flask, the precipitate washed, the washings added to the flask, and finally the silver nitrate in the second flask added also. The whole is then made up to 250 c.c. with water, mixed, filtered through a dry filter, and 50 c.c. or 100 c.c. acidified with nitric acid free from nitrous acid, and titrated with potassium thiocyanate with ferric sulphate as indicator. The following comparative results are given:

Substance.	Origin.	Required theoretically.	Gravimetric. Quantity taken.	Gravimetric. Iodide of Silver	Gravimetric. Found.	Volumetric. Quantity taken.	Volumetric. Potassium Thiocyanate required for 50 c.c. filtrate.	Volumetric. Found.
			Gramme.	Gramme.		Gramme.	C.c.	
Resacetophenone monoethyl ether $C_8H_7O_2-(OC_2H_5)$	Prepared by the author	25% OC_2H_5	0·2165	0·284	25·11% OC_2H_5	0·186	12·95	24·79% OC_2H_5
Resacetophenone monomethyl ether $C_8H_7O_2-(OCH_3)$	Prepared by the author	18·67% OCH_3	0·2515	0·36	18·87% OCH_3	0·25	12·0	18·60% OCH_3
Monoethyl-β-resorcylic acid $C_7H_5O_3-(OC_2H_5)$	Prepared by the author	24·72% OC_2H_5	0·21	0·271	24·70% OC_2H_5	0·263	12·1	24·80% OC_2H_5
Resacetophenone diethyl ether $C_8H_6O(OC_2H_5)_2$	Prepared by the author	43·26% OC_2H_5	0·2595	0·581	42·85% OC_2H_5	0·229	10·65	42·73% OC_2H_5
Dioxyethylbenzoyl formic acid $C_9H_4O_3(OC_2H_5)_2$	Prepared by the author	37·81% OC_2H_5	0·2734	0·538	37·67% OC_2H_5	0·2125	11.45	37·58% OC_2H_5
Phenacetin $C_8H_8NO(OC_2H_5)$	Commercial product	25·13% OC_2H_5	0·3445	0·450	25·01% OC_2H_5	0·3445	11·15	25·14% OC_2H_5

H. H. B. S.

Analysis of Lysol, Creolin, and Similar Preparations. H. Ditz and R. Clauser. (*Chem. Zeit.*, 1898, xxii., 732.)—An analysis of such products as lysol and carbolic soap involves the separation of phenols, hydrocarbons, and fatty acids one from another; and the present process depends on the fact that barium oleate is quite insoluble in hot and cold water, and only slightly soluble in 50 per cent. alcohol, whereas barium phenolate dissolves in 40 per cent. of its weight of water at 100° C., the orthocresylate in 150 per cent., the paracresylate in 320 per cent., while the rare metacresylate is very soluble. Barium oleate, however, being somewhat uncertain in composition, cannot be weighed as such: it is necessary again to liberate the oleic acid. Moreover, the fatty acids in lysol, etc., are not pure oleic, but contain stearic and palmitic as well, the barium salts of which are partly decomposed by water. Nevertheless, preliminary experiments have shown that working on lysol, which usually contains about 40 per cent. of fatty acids and 40 per cent. of phenols, the amount of the former recovered is only 0·5 per cent. too low, and this is a deficiency of no importance in the present investigation.

To carry out the analysis, 5 grammes of lysol are dissolved in 100 c.c. of warm water, 20 or 30 c.c. of 10 per cent. caustic soda added, and the mixture extracted two or three times with ether to remove the hydrocarbons. The ethereal solution is washed with very weak soda, and the latter combined with the bulk of the aqueous liquid; the ether is dried with solid potash, cautiously evaporated, and the residue dried over sulphuric acid to constant weight. The aqueous solution is carefully freed from ether on the water-bath, neutralized with hydrochloric acid, cooled completely (this is most important), treated with excess of barium chloride and a quantity of baryta-water roughly equivalent to the phenols present. The mixture is

filtered quickly, avoiding unnecessary exposure to the air, the precipitate washed with baryta-water, then cold and finally hot water, rinsed back into the original beaker, decomposed with 1 : 1 hydrochloric acid, and thrown on to the first filter again. The oleic acid is washed with hot weak acid till free from barium, with water till free from acid, dissolved in absolute alcohol and ether, and evaporated to constant weight at 100° C.

The filtrate from the barium precipitate may be acidified, extracted with ether, and the phenols recovered by evaporation; but it is better to operate as follows: In a convenient fraction of the acidified liquid the mixed phenols are determined by the Koppeschaar method (calling the bromine absorption calculated on the whole bulk A grammes). Another portion is extracted with ether, the solvent evaporated, and the residue dried for two or three hours at 100° to 110° C. (loss by volatilization is of no consequence in this test). It is then dissolved in caustic soda or baryta and titrated as before, calling the weight of the whole residue *a* grammes, and its bromine absorption B. The weight of the lysol originally taken for analysis being C grammes, the percentage of phenols is given by the formula:

$$\frac{100 \times A \times a}{B \times C}$$

F. H. L.

Volumetric Determination of Nitrophenol Derivatives. L. Schwarz. (*Monatshefte für Chemie*, xix., 139.)—The author has carried out experiments upon the determination of the nitrophenol derivatives by taking advantage of the liberation of free iodine resulting from the action of potassium iodide and iodate. The iodine was determined volumetrically by a $\frac{N}{10}$ sodium thiosulphate solution freshly standardized before each series of experiments was commenced. The starch emulsion was prepared as recommended by Zulkowski and Topf (*Zeit. für analyt. Chemie*, xxvi., 138) by diluting a glycerin emulsion with solution of common salt. The results show that tri- and tetra-nitro derivatives can be determined in this way without particular regard being paid to the length of time allowed for the action; but that in the case of dinitro-derivatives substitution of iodine takes place if the heating is too long continued, and for the same reason mononitro-derivatives cannot be determined in this way at all. Experiments with tribromophenol and with tetrabromfluorescin (eosin) led to the conclusion that these substances are not adapted to this method of determination.

H. H. B. S.

The Composition of the Ash of some Raw Tanning Materials. W. K. Alsop and J. H. Yokum. (*Jour. Amer. Chem. Soc.*, 1898, xx., 338-340.)—With the object of obtaining fuller information on the composition of some of the common sources of tannin, the authors have made complete analyses of a large number of samples, the average results of which are given in the subjoined table.

The method by which the tannin was determined was that of the Association of Official Agricultural Chemists, 1897. The total extract was obtained by extracting the bark with boiling water and evaporating the solution, the residue in the extractor, returned as cellulose and insoluble matter, being determined by difference. The soluble solids were the solids of the extract soluble at 18° C., and the red colouring

matter the difference between the total and the soluble solids. The non-tannins (mainly glucosides of unknown composition) consisted of the soluble solids not absorbed by hide.

The very much larger amount of ash in the oak than in the hemlock bark was due to the fact that the latter does not grow on a limestone soil.

	Chestnut Bark.	Chestnut Bark.	Hemlock Bark.	Hemlock Bark.	Quebrach Wood.	Oak-bark Extract.
No. of samples	118	117	81	50	1	1
Total extract	24·69	19·76	19·33	19·96	24·80	46·92
Total soluble solids ...	20·36	16·26	15·40	16·11	19·20	45·96
Red colouring matters ...	4·33	3·50	3·93	3·85	5·60	0·96
Non-tannins	9·81	8·21	6·74	6·44	3·64	20·29
Tannins	10·55	8·05	8·66	9·67	15·56	25·67
Moisture	10·85	9·73	11·57	13·75	11·60	53·17
Cellulose and insoluble matter	64·46	70·51	69·10	66·29	63·60	—
Ash	6·02	8·05	1·65	1·45	0·88	1·35
Carbon	0·77	0·11	0·23	0·59	0·11	1·57
Sand and silicic acid ...	2·34	3·33	—	—	0·88	1·88
Sand	—	—	1·12	3·53	—	—
SiO_2	—	—	0·78	2·84	—	—
$Al_2O_3 + Fe_2O_3$	0·08	0·86	3·07	3·86	0·64	5·59
MnO	1·26	0·33	2·26	4·09	0·10	2·21
CaO	52·63	58·24	52·52	49·35	61·27	19·15
MgO	1·18	1·77	1·98	2·92	4·56	2·38
K_2O	2·95	2·62	6·02	7·64	0·24	29·44
Na_2O	0·63	0·40	0·27	0·95	1·22	1·01
Cl	0·44	0·10	0·20	0·24	undetermined	undetermined
SO_3	0·17	0·12	0·82	1·88	1·11	2·86
P_2O_5	0·64	1·13	2·62	2·94	0·50	3·72
CO_2	36·51	31·47	27·66	19·10	28·30	undetermined
	99·62	100·49	99·55	99·93	98·93	—

C. A. M.

INORGANIC ANALYSIS.

Estimation of Lead in its Ores. L. Schneider. (*Oesterr. Zeits. Berg. u. Hüttenw.*, 1898, xlvi., 431; through *Chem. Zeit. Rep.*, 1898, 212.)—The author has investigated the influence of several substances upon the degree of insolubility of lead sulphate. He finds that tartaric, and more particularly nitric, acids raise its solubility in water; and that, although the presence of sulphuric acid, tartaric acid, or ammonium sulphate diminishes the solvent action of the nitric acid, yet lead cannot be completely precipitated unless the latter forms less than 1 per cent. of the liquid. He suggests the following process for the analysis of lead ores: 1 gramme is boiled for ten or fifteen minutes with 5 c.c. of strong hydrochloric acid, when 5 c.c. of strong nitric acid are added, and the mixture heated till the brown vapours

disappear. The lead salt is dissolved in 10 grammes of tartaric acid and 25 c.c. of water, and treated with 25 c.c. of strong ammonia. The insoluble residue consists only of quartz and barium sulphate. The filtrate, diluted to 300 c.c., is warmed with 50 c.c. of 1 : 1 sulphuric acid, the precipitate washed with 1 per cent. acid, dried and weighed. If all these conditions are adhered to, there is no necessity to drive off the nitric acid, because in the final filtrate and washings, which amount to about 500 c.c., 0·003 gramme of lead sulphate always remains in solution, and the obvious correction can easily be made. F. H. L.

Volumetric Estimation of Lead Peroxide by Means of Arsenious Acid. C. Reichard. (*Chem. Zeit.*, 1898, xxii., 774.)—Although lead peroxide is not attacked by an aqueous or ammoniacal solution of arsenious acid, in strong caustic soda solution at the boiling-point it is quantitatively reduced to monoxide: $2PbO_2 + As_2O_3 = 2PbO + As_2O_5$. Since the substance is largely used as an oxidizer in the manufacture of dyes, the percentage of available oxygen it contains is often the most important factor, and to save calculation, therefore, the analysis may be conducted exactly as follows: The peroxide is brought to a fine powder, being preferably passed through a cloth; 0·1 gramme is weighed out, and introduced into a known excess of a solution of arsenious acid in concentrated caustic soda containing 0·0099 gramme of As_2O_3 (= 0·0016 gramme of oxygen) per c.c. The whole is boiled with further additions of highly concentrated alkali till the liquid is colourless or but faintly yellow. It is then diluted, acidified with sulphuric acid, and the amount of arsenious acid remaining estimated by titration with permanganate. If preferred, the neutral solution may be filtered, treated with sodium bicarbonate, cooled, and similarly titrated with iodine and starch. F. H. L.

Examination of Alloys containing Lead and Antimony. A. Fraenkel. *Mittheil. techn. Gew. Mus. Vienna*, 1898, viii., 334; through *Chem. Zeit. Rep.*, 1898, 229.)—Alloys which contain lead, copper, and phosphorus as well as tin and antimony are attacked with nitric acid, the residue fused with caustic soda in a silver crucible, the melt extracted with water, diluted with one-third its volume of alcohol, and filtered after standing for twenty-four hours. The filtrate (F) contains sodium stannate and phosphate, and the whole of the lead undissolved by the nitric acid at first; the residue (R) contains the antimony and copper. In (F) the lead is precipitated with sulphuretted hydrogen and determined as usual; the filtrate is treated with sulphuric or acetic acid to throw down tin sulphide, which is converted into oxide and weighed as such. The filtrate from the tin is evaporated and the phosphoric acid estimated. (R) is dissolved in hydrochloric and tartaric acids, and precipitated with sulphuretted hydrogen; the antimony sulphide is dissolved by digestion with ammonium sulphide, filtered from the copper, thrown down together with some sulphur by means of acid, collected on a tared filter, and the antimony determined in a portion by ignition in a current of carbon dioxide. The original nitric acid solution is investigated in the ordinary fashion. F. H. L.

The Determination of Lead in Alloys. W. E. Garrigues. (*Journ. Amer. Chem. Soc.*, 1898, xx., 508-510.)—The following method is based on the fact that lead chromate is insoluble in ammonia, whilst copper chromate is readily soluble. Where applicable, it is much more rapid than precipitation of the lead as sulphate.

The solution of the lead and copper in nitric acid is mixed with potassium bichromate in excess, followed by a large excess of ammonia. The liquid is heated until the supernatant liquid becomes clear, and the precipitate of lead chromate collected in a Gooch crucible containing a single disc of filter-paper as the filtering medium, washed with dilute ammonia, hot water, and alcohol, and dried in the water-oven.

This method is essentially a separation of lead from copper and zinc; but when antimony, bismuth, or iron are present, the sulphate method must be employed. In determining lead in the presence of antimony, it is possible to prevent the small quantity left in the filtrate from the tin from being precipitated with the lead sulphate by adding a large amount of sulphuric acid, diluting sparingly, and filtering as rapidly as possible.

A rapid modification of the sulphate process, in which the lead is obtained without previous removal of tin, is described in the form of an account of an experiment:

Half a gramme of solder, containing, according to the sulphate and chromate determinations, 58·83 and 58·80 per cent. of lead respectively, was oxidized with nitric acid, 20 c.c. of strong sulphuric acid added, and the liquid evaporated until dense fumes appeared. The metastannic acid was completely dissolved. After adding 80 c.c. of cold water, the solution was well stirred and the lead sulphate collected on a Gooch's filter. The quantity of lead thus found was 59 per cent.

The filtrate was made alkaline with ammonia, and sulphuric acid added until the liquid turned methyl orange decidedly red. The liquid was boiled, and the voluminous white precipitate collected on a filter, ignited in a strong blast, and weighed as stannic oxide. The amount of tin calculated from the result was 41·1 per cent.

If more than 80 c.c. of water were used for the dilution, or if the solution was left on the water-bath for an hour or two, the tin began to precipitate slowly.

C. A. M.

Estimation of Nickel in the Presence of Iron. B. Neumann. (*Chem. Zeit.*, 1898, xxii., 731.)—Five grammes of steel borings are dissolved in dilute sulphuric acid, and the carbon and iron oxidized with bromine, nitric acid, or preferably hydrogen peroxide. The solution is brought into a 500 c.c. flask, ammonium sulphate added, the iron precipitated with ammonia, the liquid boiled, excess of ammonia introduced, and after cooling diluted to the mark. One hundred c.c. (= 1 gramme) are filtered off, diluted slightly, warmed and treated with a current of 1 or 2 ampères at 3·4 to 3·8 volts for one-and-a-half to two hours. The space occupied by freshly-precipitated ferric hydrate is not known, but in ordinary steels the error thus caused will not exceed 0·5 per cent. of the nickel obtained. In steels rich in nickel, where only 0·5 gramme is taken for electrolysis, it will be about 0·2 per cent.; and in nickel

ores, etc., containing 50 or 60 per cent. of iron, not more than 0·2 or 0·3 per cent. Nickel ores are attacked first with hydrochloric acid, then with nitric, and evaporated with sulphuric acid till white fumes appear. The heavy metals are thrown down with sulphuretted hydrogen, excess of the reagent removed by heat, the liquid oxidized with bromine or hydrogen peroxide, and 100 c.c. of the solution (= 1 gramme of material) treated as before. Cobalt remains in the liquid, and is deposited with the nickel.

One sample of nickel-steel which gave 4·12 per cent. of Ni when the iron was removed by fourfold precipitation gave 4·02 per cent. of Ni by the author's process; electrolysed in presence of the ferric hydrate, the yield was 4·00 per cent. (corrected). A specimen of nickel ore examined by the author's method gave 6·02 per cent. of Ni + Co; four precipitations of the iron gave 6·15 per cent. of Ni + Co; electrolysis without removal of the iron gave 5·98 per cent. of Ni + Co (corrected); Rothe's process (extraction of the hydrochloric acid solution with ether, to dissolve iron and cobalt) gave 5·60 per cent. of Ni. The proportion of cobalt, specially estimated with nitroso-β-naphthol, was 0·50 per cent. F. H. L.

The Use of Hydrofluoric Acid in the Determination of Manganese in Iron and Ores. A. P. Ford and I. M. Bregowsky. (*Journ. Amer. Chem. Soc.*, 1898, xx., 504-506.)—In order to avoid the evaporation which is necessary when hydrofluoric acid is used in the determination of manganese in high silicon pig-irons, the authors have made experiments on the direct use of the acid with eventually successful results. The iron is dissolved, and the manganese precipitated with potassium chlorate. As soon as the precipitation is complete a few drops of hydrofluoric acid (the quantity depending on the amount of silicon present) are added, and the liquid boiled until the hydrofluoric acid is expelled. A little more potassium chlorate is then added (about 1 gramme), and the liquid concentrated as much as desired. The solution filters rapidly, and the graphitic carbon will be found to be almost completely oxidized owing to the removal of the enveloping silica.

Similarly, in the case of manganese ores a few drops of hydrofluoric acid are added *after* the precipitation of the manganese with potassium chlorate, and if the first addition does not clear the solution in a minute or two, a few more drops are added. With a little practice, the amount required can be judged by the amount of floating gelatinous silica.

As to the action of the acid on the beakers, the authors state that, though they become opaque, their life is but little diminished. It is, however, advisable to keep a number of beakers apart for manganese determinations only. C. A. M.

Aluminium used as an Electrode. G. J. Hough. (*Journ. Amer. Chem. Soc.*, 1898, xx., 302, 303.)—The author states that aluminium can replace platinum as an electrode to a limited extent. It can be used as the cathode with nitric acid solutions without being affected, but cannot be used as the anode, for at that pole it is readily oxidized and dissolved. It is attacked by the free acid in sulphate or chloride

solutions, but can be employed when these are converted into the double oxalate of potassium or ammonium, as recommended by Classen. It is not attacked by cold solutions of potassium cyanide or oxalate, even under electrolytic action. Not being acted upon by hot or cold nitric acid, the metals deposited on it should readily be dissolved, but the author has not yet tested its applicability thoroughly and minutely, except in the case of copper analysis. Being so much lighter than platinum, its use reduces the chance of error in weighing the deposited metal, while its cheapness is a further advantage. C. A. M.

Detection of Normal Carbonate in Bicarbonate of Soda. N. Kubli. (*Arch. Pharm.*, 1898, ccxxxvi., 321; through *Chem. Zeit. Rep.*, 1898, 228.)—A 0·1 per cent. solution of quinine hydrochloride is not precipitated by sodium bicarbonate unless more than 2 per cent. of normal carbonate is present. To apply the test, 3 grammes of bicarbonate are dissolved in 50 c.c. of water at a temperature of 5° or 6° C., without agitation, in a closed vessel. On adding an equal volume of the quinine reagent, the mixture should remain clear. F. H. L.

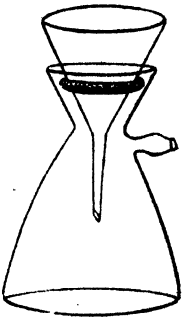
Volumetric Estimation of Nitric Acid. W. Ackermann. (*Chem. Zeit.*, 1898, xxii., 690.)—This process depends on the reduction of nitrates to ammonia by means of ferrous hydrate. One gramme of potassium nitrate, 30 c.c. of caustic soda (specific gravity 1·33), and 40 grammes of crystallized ferrous sulphate, are placed in a 500 c.c. flask with 160 c.c. of water; a small quantity of reduced iron is added to assist the boiling, and the liquid is distilled into standard acid in the usual fashion. The operation proceeds quietly, and is generally finished in half an hour; but the complete evolution of the ammonia may easily be ascertained by removing the beaker containing the acid, washing the outside of the condensing tube, and testing the next 30 or 40 drops of distillate with litmus-paper. Should this prove to be still alkaline, more water may be added to the flask, and the distillation continued slowly; but as it is a sign that the reduction has not been successful, it is perhaps better to repeat the whole analysis. Blank experiments are necessary to estimate the amount of nitric acid in the several reagents, and in order to keep the correction as small as possible, the ferrous sulphate should be prepared from iron wire and pure acid, and good commercial caustic soda employed instead of the "pure" article. The above-mentioned proportion of water may be doubled or trebled if desired, but larger quantities tend to imperfect reduction, because on prolonged boiling and before suitable concentration is reached the ferrous hydrate loses its activity. The weight of caustic soda should be kept as specified; that is to say, it should form only a slight excess over the amount needed to decompose the ferrous sulphate.

Alkali chlorides, sulphates, and acetates do not interfere with the process; tartrates hinder the reaction; and phosphates, as existing in artificial manures, involve the following modification: The solution of the fertilizer is mixed in the distilling flask with sufficient calcium chloride solution of known strength to convert the whole of the soluble phosphorus into tribasic phosphate; the alkali is then dropped in with constant agitation, until an excess equivalent to 2 c.c. of the calcium chloride is attained; the ferrous sulphate and metallic iron are next added; the

liquid is made up to 170 c.c. (to allow for the extra viscidity caused by the calcium phosphate), and the analysis is finally conducted as before.

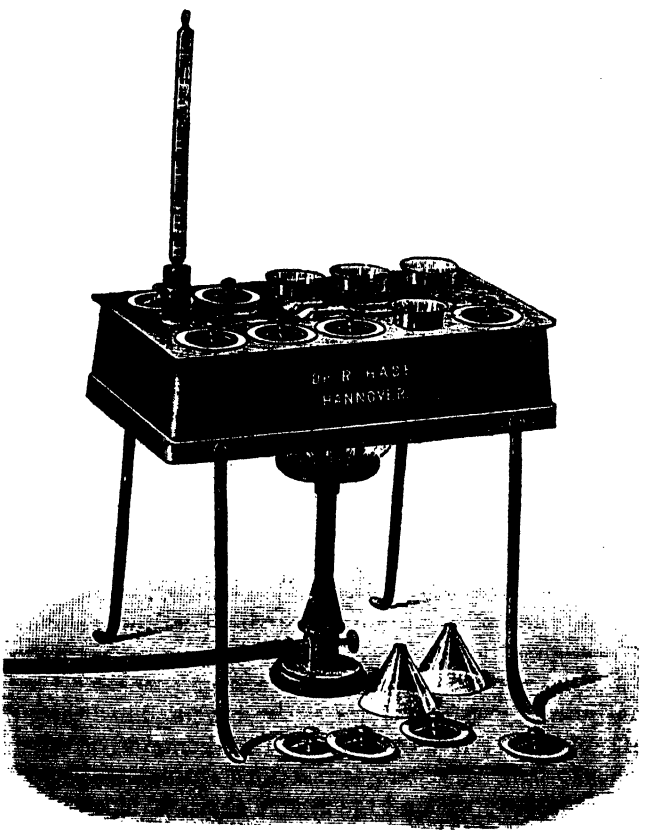
Figures quoted by the author show that, in working on 1 gramme of saltpetre under ordinary conditions, the variations in the end results will not exceed 1 or 2 milligrammes. F. H. L.

APPARATUS.



A New Vacuum Filter Flask. R. Walther. (*Pharm. Central. H.*, 1898, xxxix., 550; through *Chem. Zeit. Rep.*, 1898, 209.) — The construction of this apparatus is shown in the sketch, the shaded portion being a hollow (pneumatic) rubber ring filled with air, which fits any flask or funnel indifferently, and makes a tight joint by its own compression. F. H. L.

Apparatus for the Determination of the Reichert - Meissl Number of Butter. M. Siegfeld. (*Chem. Zeit.*, 1898, xxii., 738.) — In the Milchwirthschaftliche Institut at Hameln a pipette is used for measuring out the quantity of butter-fat required for this process, which, when manipulated uniformly, always delivers between 4·967 and 5·045 grammes. The maximum variation, therefore, corresponds to a difference of 0·2 or 0·3 in the Reichert - Meissl number; and this is quite insignificant in comparison with other sources of error. For the filtration of the fat and for keeping the pipettes warm a special water-bath, as illustrated herewith, is employed. The thermometer has a red mark at 50° C. to make it more conspicuous; but for ordinary purposes the temperature needs only to be maintained between 45° and 55° C. F. H. L.



REVIEWS.

A Short Course of Inorganic Qualitative Analysis for Engineering Students. By J. S. C. Wells, Ph.D., Instructor in Analytical Chemistry, Columbia University. New York: John Wiley and Sons. London: Chapman and Hall. 1898.

The object of this work is to give a "short but thorough" course in inorganic qualitative analysis for the use of students who have but a limited time to devote to the subject. There is no doubt that the student who may use Mr. Wells' book with zeal and discretion will necessarily gain a valuable knowledge of elementary qualitative analysis, but the work is no royal road to an understanding of the subject; and although excellent in many respects, is not without serious defects. To begin with some of its excellences—equations are freely used, the tables of separation are well printed, arranged, and expressed, and the tables of reactions representing skeleton schemes of separation (using formulæ only) form a valuable and somewhat original feature. For the rest, it differs but little from the innumerable works of this kind, and lacks the masterly treatment which we find in such manuals as those of Clowes and Coleman, Tilden, or Thorpe and Muir. It is admittedly based on Fresenius, who has been curtailed and improved upon in a somewhat wayward fashion. The descriptions of reactions are too lengthy and involved, and there is much unnecessary waste of space in text and tables, so as to leave an impression that the book would be equally useful if reduced to 150 pages, instead of the 300 of which it consists at present.

H. W. H.

A Laboratory Guide in Qualitative Chemical Analysis. By H. C. Wells, M.A., Professor of Analytical Chemistry and Metallurgy in the Sheffield Scientific School of Yale University. New York: John Wiley and Sons. London: Chapman and Hall. 1898.

This work is the printed presentment of a method of instruction which has been used by the author for fourteen years. Fresenius, who might, indeed, be called the primogenitor of analytical text-books, has again been largely drawn upon; but changes have been made where the author has considered them "advantageous," and a "few novelties" have also been introduced. One of these is a rearrangement of Fresenius' groups, which seems liable to render confusion worse confounded.

The work has but little to recommend it, and its sphere of usefulness might be greater if it were much condensed. The main idea seems to be an attempt to make the student evolve analytical methods out of his own inner consciousness by a series of elaborate exercises, which are by no means of a practical character. Take, for instance, the first exercise, where the pupil is directed to find by experiments which, out of twenty-seven solutions (of the common metals), yield a precipitate with hydrochloric acid. The simple reactions of this group are dealt with at inordinate length, and similarly with all the groups through fifty-six long pages. Blowpipe tests are not considered, as "they are usually studied in connection with determinative mineralogy"; but Fresenius may be consulted. Next following we have directions

for making reagents which are used in the first part. The ensuing chapter, on ionization and ions, is far too brief to be of any real value or interest; and after various chapters on the classification of salts and chemical equations, etc., we are finally, on p. 101, introduced to the reactions of the metals and acid radicles!

The most original feature of the work consists in twenty pages of chemical labels at the end, arranged so as to be torn out for use in the laboratory. H. W. H.

PRACTICAL ORGANIC CHEMISTRY: THE DETECTION AND PROPERTIES OF SOME OF THE MORE IMPORTANT ORGANIC COMPOUNDS. By S. RIDEAL, D.Sc., F.I.C. Second edition. London: H. K. Lewis. 1898.

Many students, and not a few teachers, will welcome the second edition of this useful and able little volume, which fills a special gap in analytical text-books. It deals with a definite class of compounds in a direct and succinct fashion, which is extremely refreshing in comparison with the two last-mentioned works, with which it is only fair to say it has nothing in common. The reactions of several important organic compounds have been added, and note has been taken of alterations in the new British Pharmacopœia, but the size of the book has not been unduly increased. On one point only can we bring ourselves to disagree with Dr. Rideal, and that is as regards the omission of any scheme of analysis; such a scheme is, we think, much needed. A summary of distinctive tests for each group is, however, still included, and forms a basis on which an earnest student may work out a scheme for himself. Although the work is specially adapted for the London University and other examinations, it will without doubt be appreciated by a wider circle of scientific workers.

H. W. H.

LABORATORY TEXT-BOOK FOR BREWERS. By LAWRENCE BRIANT. Second edition. (London: Fell and Briant.) Price 10s. 6d. net.

The first edition of this useful work, which was published in 1884, was for a long time the standard book on the analysis of the materials used in the brewery; but as time went on, and in the meantime this department of chemistry progressed somewhat rapidly, the work naturally fell behind the times. The new edition, which has been promised for a very long time, effectually disposes of this drawback, for it has been thoroughly brought up to date, and to do this effectually has necessitated the re-writing of the whole book. The new edition possesses all the good qualities of the old, such as simplicity and clearness of style, and the student is greatly assisted by the examples appended to the description of the various analyses. A few statements have crept in which will need correction in a future edition, of which the following are examples: P. 4, "Ungelatinized starch is not acted upon by diastase"; p. 20, "Levulose oxidizes into saccharic acid by contact with dilute nitric acid"; p. 27, "Amido-acids are formed when amides are heated with a mineral acid." However, these are trifles when compared with the otherwise excellent character of the information supplied, which will be found most useful to brewing analysts and students of brewing chemistry. W. J. S.

TECHNICAL MYCOLOGY: THE UTILIZATION OF MICRO-ORGANISMS IN THE ARTS AND MANUFACTURES. By Dr. FRANZ LAFAR, with a Preface by Dr. EMIL C. HANSEN. Translated by CHARLES T. C. SALTER. Vol. I.: Schizomycetic Fermentation. (London: Charles Griffin and Co.) Price 15s.

This is the first time that mycology in its practical application to the arts and manufactures has been treated in a thorough and exhaustive manner. Such a book as this was urgently needed, for in recent years the subject on which it treats has made enormous advances; it is only when the whole of the instances are collected together that we realize how numerous the industries are in which micro-organisms play an important part.

The introduction is divided into three parts; the first of these treats on the theory of spontaneous generation, and gives a historical résumé of the various views on this subject which have dominated the scientific world from time to time. The author is of opinion that, though abiogenesis is yet unproven, it may not be impossible. The next division contains a short and similarly historical summary of the principal views which have been advanced in explanation of the phenomena of fermentation. Dr. Lafar defines this process as "a decomposition or transformation of substances of various kinds induced by the vital activity of fungi." The third division treats of the position of the organisms of fermentation in the botanical system. We now come to the body of the book, which is divided into sections. The first of these is devoted to a description of the general morphology and physiology of the schizomycetes; Section II. to their general biology and classification; Section III. treats on the methods of sterilization and for obtaining pure cultures; the following section on the behaviour of the chromogenic, photogenic, and thermogenic bacteria. The remaining five sections are mainly devoted to the consideration of those micro-organisms which are immediately concerned in, or affect, processes carried out in manufacturing operations, such as the "retting" of flax, souring of cream, the fermentation of the liquors used in tanning, the ripening of cheese, the manufacture of vinegar, and many others. From this some idea may be gained of the comprehensive nature of the work, which is, with reference to these subjects, a perfect mine of wealth. Without doubt the book is indispensable to all who are engaged in any of those industries in which bacterial fermentations are concerned, and for the scientist it forms a valuable work of reference; so good is it that we look forward with something like impatience for the appearance of the second volume, which will treat on the Saccharomycetes and the fermentations they induce.

The book is well printed, the illustrations excellent, and, altogether, it is got up in handsome style. The translator, Mr. Salter, is to be congratulated on the excellent manner in which he has accomplished his task. W. J. S.

THE ANALYST.

DECEMBER, 1898.

PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS.

THE monthly meeting of the Society was held on Wednesday evening, November 2, in the Rooms of the Chemical Society, Burlington House, the President (Dr. Bernard Dyer) occupying the chair.

The minutes of the previous meeting were read and confirmed.

The following gentlemen were proposed for election as members of the Society: Mr. J. F. H. Gilbard, 8, Glaskin Villas, Lea Bridge Road, London, N.E., Gas Examiner; Mr. Martin Priest, Assayer to the Imperial Mint, Nanking, China; and Mr. Lester Reed, F.I.C., 19, South Park Hill Road, Croydon, Public Analyst for the Borough of Croydon.

Mr. G. W. Slatter, A.R.C.Sc., F.I.C., was elected a member; and Mr. A. J. Bull was elected an associate.

The following papers were read and discussed: "A Curious Meat-Preservative" and "A Method for the Quantitative Separation of Acetic and Valeric Acids," by Alfred C. Chapman. "Automatic Pipettes and Burettes" and "The Effects of Recent Drought on the Quality of Milk," by A. W. Stokes. A paper entitled "Contributions to the Chemistry of Drying Oils, with a Method for the Examination of Linseed Oil," by Otto Hehner and C. A. Mitchell, B.A., was then read, but its discussion, on account of the lateness of the hour, was postponed.

A CURIOUS MEAT-PRESERVATIVE.

BY ALFRED C. CHAPMAN, F.I.C.

(Read at the Meeting, November 2, 1898.)

SOME time ago I received for analysis an antiseptic solution intended for the preservation of meat. The liquid, which was colourless, possessed a mixed odour of sulphurous and benzoic acids, and proved on examination to have a somewhat remarkable composition. The analysis, which was only qualitative, revealed the presence of the following substances: aluminium sulphate, sodium chloride, sodium nitrate, sulphurous acid, chloral hydrate, benzoic acid, and a small quantity of iodine, apparently existing as hydriodic acid. The alum was evidently intended to exert its astringent effect upon the fibre of the meat, whilst the remaining constituents (with the exception, perhaps, of the chloral) are, of course, well known to be possessed of marked preservative and antiseptic properties. Both the chloral and the benzoic acid would doubtless be volatilized during the cooking of the meat, and

this fact probably suggested the use of benzoic instead of the more active salicylic acid. In the course of my practice I had never before encountered chloral hydrate as a constituent of a food-preservative, and I thought that a short note calling attention to its use for such a purpose might not prove uninteresting to the members of this Society.

DISCUSSION.

DR. RIDEAL said that preservatives of this kind were almost invariably of French origin. He had met with several, the composition of which was similar in character to that of Mr. Chapman's sample. Chloral hydrate was present in some, but not in all. The main idea of the manufacturers of such mixtures seemed to be to use a large number of different ingredients, apparently with a view to baffling analysts.

CONTRIBUTION TO THE CHEMISTRY OF DRYING OILS; WITH A METHOD FOR THE EXAMINATION OF LINSEED OIL.

BY OTTO HEHNER AND C. A. MITCHELL, B.A.

(Read at the Meeting, November 2, 1898.)

OUR present knowledge of the composition and constitution of the fluid fatty acids occurring in natural fluid glycerides dates back no further than the year 1888, when K. Hazura, together with a number of collaborateurs, published a series of exceedingly important papers in the *Monatshefte für Chemie*. Up to that time the fluid portion of oils, drying and non-drying, was described as olein.

Hazura showed that when fluid fatty acids obtained from various oils were oxidized in alkaline solution with potassium permanganate, a number of different hydroxy-derivatives were produced. He isolated and described the characteristics of these compounds, and concluded from his investigations that the unsaturated fatty acids from fixed oils take up as many hydroxyl groups as there are free valencies, yielding saturated hydroxy-fatty acids containing the same number of carbon atoms as the original molecule. He also showed that the unsaturated fatty acids in question combined directly with bromine, as many bromine atoms being fixed as there are free valencies. He thus differentiated between acids of different degrees of non-saturation, and supplied a scientific explanation of the Hübl iodine value.

We give in a tabular form a statement of his results:

TABLE I.

Original Acid.	Bromo-compound.	M.P.	Oxidation Product.	M.P.
Oleic ($C_{18}H_{34}O_2$)	Dibrom-stearic acid $C_{18}H_{34}Br_2O_2$	Liquid	Dihydroxy-stearic acid $C_{18}H_{34}(OH)_2O_2$	137° C.
Linolic ($C_{18}H_{32}O_2$)	Tetrabrom-stearic acid $C_{18}H_{32}Br_4O_2$	114–115° C.	Sativic acid $C_{18}H_{32}(OH)_4O_2$	173° C.
Linolenic ($C_{18}H_{30}O_2$)	Hexabrom-stearic acid $C_{18}H_{30}Br_6O_2$	177° C.	Linusic acid $C_{18}H_{30}(OH)_6O_2$	203° C.
Isolinolenic ($C_{18}H_{30}O_2$)	—	—	Isolinusic $C_{18}H_{30}(OH)_6O_2$	173–175° C.

From the yields of the hydroxy-derivatives, which, as will be seen from the following tables, were very far from quantitative, Hazura and Grüssner made an approximate estimation of the composition of the unsaturated acids of a number of oils:

TABLE II.

	100 Grammes of Liquid Fatty Acids yielded:				
	Linseed.	Hemp.	Nut.	Poppy.	Cotton.
Dihydroxy-stearic acid ...	1·2	4·0	2·5	8·5	23·0
Sativic acid	6·5	24·0	25·0	18·5	31·5
Linusic + isolinusic acids ...	20·3	2·5	2·0	0·3	—
	28·0	30·5	29·5	27·3	54·5

TABLE III.

	Approximate Percentage Composition of Liquid Fatty Acids.				
	Linseed.	Hemp.	Nut.	Poppy.	Cotton.
Oleic acid	5	15	7	30	40
Linolic acid	15	70	80	65	60
Linolenic / Isolinolenic acids	15 / 65	15	13	5	0

It is obvious from the numbers given in Table II. that there must have been a considerable destruction of the first products of oxidation, especially of those derived from the least saturated acids; Hazura, in fact, obtained a number of further oxidation-products, indicating the rupture of the molecule. Nevertheless, it may be interesting to compare Hazura's estimates and the iodine values deduced therefrom, with the actual iodine numbers recorded for the fatty acids from the several oils, bearing in mind that the latter are for the total free fatty acids, while Hazura's numbers are for the unsaturated acids only.

TABLE IV.

	Linseed.	Hemp.	Nut.	Poppy.	Cotton.
Iodine number calculated from Hazura's results	251	181·5	187	158	145
Iodine value of total acids ...	160-180	141	145	139	113

The calculated iodine value of oleic acid is 90; of linolic acid, 181·4; and of linolenic and isolinolenic acid, 274·1.

With regard to isolinolenic acid, Hazura and Grüssner inferred its existence solely from the oxidation-products formed; they did not obtain a bromo-derivative, and hence did not isolate the acid.

After making allowance for the presence of saturated fatty acids, it will be seen that there is an approximate correspondence between the calculated and the actual figures in the case of all but the linseed oil, which is said to contain about 15 per cent. of solid acids.

We have repeated some of Hazura's work, and have prepared some, at least, of the derivatives discovered by him, paying particular attention to the bromine-derivatives.

On adding a sufficiency of bromine to an ethereal or acetic acid solution of linseed fatty acids, kept at a temperature of about 5° C., an abundant precipitate immediately forms. Part of this is exceedingly insoluble, while a part can be washed away with ether. The insoluble portion is the hexabrom-derivative of Hazura, although the composition of the substance does not exactly correspond with the formula, even after extraction for days with ether.

Thus, in four separate experiments we obtained compounds with the following percentages of bromine :

	Found.	Theoretical.
1.	61·38	63·31
2.	61·47	
3.	61·80	
4.	61·64	

We found the melting-point from 180° to 181° C., while Hazura gives 177° C. We determined the percentage of bromine-derivative yielded by a considerable number of samples of linseed oil fatty acids, and found them to vary from 20 to 26 per cent. Hazura obtained from 100 parts of the fluid linseed oil acids 40 of hexabromide, having a melting-point of 177° C. We believe that his precipitates must have included a considerable proportion of tetrabromide, seeing that it is difficult to entirely wash the latter out, as the above analyses of the persistently washed products show.

After the hexabromide has been filtered off, precipitates are obtainable which contain upwards of 50 per cent. of bromine, the tetrabromide formula requiring 53·32 per cent. of bromine. The part remaining in solution, when recovered by the evaporation of the solvent, consists of a semi-solid mass, evidently a mixture of dibromide and tetrabromide. This we found to contain 45 per cent. of bromine, the pure dibromide requiring 36·18 per cent. The tetrabromide of Hazura can be much more readily obtained from maize oil fatty acids than from linseed. But here, again, the actual percentage of bromine, even after numerous crystallizations from alcohol, falls somewhat short of the theoretical amount. Our product contained 51·97 per cent. of bromine, against a theoretical 53·32 per cent. The melting-point was 113·4° C. (corr.), against Hazura's 114° to 115° C.

The dibromide we have not been able to obtain in a pure condition.

On boiling the hexa- and tetra-bromine compounds with alcoholic potassium hydroxide the bromine is entirely eliminated. Theory requires for complete elimination of the bromine from the hexa-compound 51·8 per cent. of potassium hydroxide. In four separate determinations 51·33, 51·61, 51·50, and 51·5 per cent. of potassium hydroxide were consumed. On liberating the free acids from the resulting alkali salts no crystallizable substances could be obtained, the products being viscid yellow oils, which did not dry after being exposed to the air for a week, and which were, therefore, neither the hydroxyacids, which result from oxidation by permanganate, nor the less saturated products which would have resulted from an elimination of 6HBr from the molecule. At the same time, the oil absorbed iodine from Hübl's solution to the extent of 61 to 63 per cent. After the titration of the excess of iodine added, the solutions rapidly became blue again from the further liberation of iodine. On re-bromination hydrobromic acid was evolved, and the original hexabromide was not reproduced. In this particular it behaved like the acids from boiled linseed oil.

The bromine can likewise be completely removed from the dibromides and tetrabromides by boiling with alcoholic potassium hydroxide, and the acids thus obtained are also oily fluids with low iodine absorption.

Hazura, by reduction of the hexa-bromide with zinc and hydrochloric acid, prepared linolenic acid, which, however, owing to its oxidizable nature, could not be obtained pure and with the theoretical iodine value. He found 245, as compared with the theoretical 274·1. We have also made efforts in this direction, taking every precaution to keep away oxygen, yet were not more successful, since the iodine absorption of our linolenic acid only reached 241·8. The acid obtained was a nearly colourless oil of a specific gravity 0·9228 at 15·5° C. (compared with water at the same temperature) which absorbed oxygen from the air with great rapidity and quickly became dark brown. On dissolving this linolenic acid in acetic acid and adding bromine, the hexabromide was produced, but not quantitatively, only 46 to 50 per cent. of the yield being obtainable; dense fumes of hydrobromic acid were given off. With the advance of oxidation in a linseed oil the yield of hexabromide decreases with the decrease in the iodine value. A linseed oil with an original iodine value of 160·7, with a yield of hexabromide of 22·5 per cent. (probably not quite pure hexa-compound) on the fatty acids, was heated on the water-bath for a day and air blown through at intervals. The iodine value of the fatty acids was 147·3, and the yield of insoluble bromo-compound obtained under precisely the same conditions as before was 16·7 and 15·9 per cent. respectively in two determinations. A sample of boiled linseed oil, with an iodine value of 115·2, gave absolutely no precipitate on treatment with bromine, but hydrobromic acid was evolved. It appears probable to us that the amount of hydrobromic acid produced will be found to be a measure of such oxidation as occurs during the "boiling" of linseed and other oils. It will be remembered that McIlhiney (ANALYST, xix., 141) has already made experiments, from which he deduces a method for the determination of resin-acids in linseed oil, based upon the elimination of hydrobromic acid on bromination.

We have also submitted the unsaturated fatty acids obtained from maize oil to oxidation with alkaline permanganate, and are in a position to entirely confirm Hazura's statements as to the composition and properties of sativic acid, except that the melting-point of our purest substance was 174°, instead of 173° as found by him.

Acting upon the fatty acids liberated from other oils with bromine, we found that almond oil, with an iodine value of 95·68, gave no solid product of any kind. Mixtures of almond and linseed oil acids yielded amounts of insoluble bromide corresponding very approximately to the percentage of the linseed oil acids present, provided the operations were always carried out under precisely the same conditions as to amount of acids taken, temperature, solvent used, and washing. Quantities of from 0·2 to 0·3 grammes of the fatty acids were dissolved in 10 c.c. of acetic acid and the solution cooled to 5° C. in a corked flask. Bromine was then added drop by drop until a permanent bromine colour was observed. After standing for three hours the liquid was filtered through a Soxhlet tube containing asbestos, and washed with chilled acetic acid, alcohol, and ether, in successive portions of 5 c.c. each. The residue, which was white, was dried in the water-oven to constant weight. Attempts to use

larger quantities of fatty acid were unsuccessful, owing to the difficulty of filtration and washing until colourless in a Soxhlet tube.

TABLE V.

Acids used.	Linseed Oil. Per Cent.	Insoluble Bromide, Per Cent.	Linseed Oil Acid calculated from Bromide.
Linseed oil	100	21	
Almond oil	0	0	0
Linseed + almond ...	7·9	1·62	7·7
,, ,, ...	8·7	1·97	9·3
,, ,, ...	20·2	4·24	20·1
,, ,, ...	37·5	7·53	35·3
Poppy oil	0	0	0
Poppy + linseed	18·3	3·35	16
,, ,, ...	37·3	8·17	38·9
Cotton-seed oil	0	0	0
Cotton-seed + linseed ...	43·3	8·43	40·1
,, ,, ...	60·1	11·6	55·2

It need hardly be said that poppy-seed and cotton-seed oil acids also furnish the tetrabromide, which, however, comes down more slowly than the hexabromide, and is more or less completely removed by washing the deposit. The deposit from poppy-seed oil acids melted at 112° C., and that from cotton-seed oil acids at 113° C.

Although the results stated in the preceding table are fairly satisfactory, yet the method is so empirical as not to commend itself. Moreover, when larger quantities of acids are operated upon, tetrabromide is apt to come down simultaneously with the hexa-compound, and to wash this free from the former considerable quantities of solvent are necessary, in which the hexabromide is not completely insoluble.

A more reliable method is to obtain the mixture of solid compounds, washing this but slightly in order to remove readily soluble dibromide, and then to determine the percentage of bromine in the dried and weighed precipitate. From the amount of bromine found it is easy to calculate the relative proportion of the hexabromide in the mixture by means of the formula

$$\frac{63{\cdot}3\,x}{100} \times \frac{(100-x)53{\cdot}3}{100} = m$$

or $10\,x = 100(m - 53{\cdot}3)$ or $x = 10(m - 53{\cdot}3)$, in which m equals the percentage of bromine found, x the required percentage of hexabromide, and 63·3 and 53·3 the respective percentage of bromine in the pure hexa- and tetra-compounds.

To illustrate this mode of working an outline of an experiment on linseed oil acids may be described. About 2 grammes of linseed oil acids (which by the method used in the experiments given in Table V. yielded from 20 to 23 per cent. of insoluble bromides) were brominated, but the deposit was washed with a far larger amount of acetic acid and ether than previously mentioned. By this washing the amount of deposit was reduced to 13 per cent., which melted at 179° C., and was therefore practically pure hexabromide. The filtrate and washings were allowed to concentrate spontaneously, whereby a further crystalline deposit amounting to 8·16 per cent. was

obtained. This deposit contained 58·8 per cent. of bromine, which, calculated by the formula given above, corresponds to 4·48 per cent. of hexabromide. Hence, the total percentage of hexabromide equalled 17·48. The filtrate from the second precipitate did not yield any further solid deposit, but only a heavy yellow oil.

This mode of operating may afford a solution of the problem of determining the relative percentages of oleic, linolic and linolenic acids in a mixture of fluid fatty acids, provided the saturated fatty acids are first separated. Further experiments on this important point will be carried out.

The idea naturally suggests itself to obviate the separation of the acids from oils and to operate upon the glycerides instead. When an oil, the fatty acids of which give insoluble bromine-compounds, is dissolved in ether or other suitable solvent, and bromine is added, there is an immediate precipitate produced, which is much more insoluble than the acid hexabromide; and consequently can be washed readily and efficiently. The precipitate can be collected either in a Soxhlet tube, if the quantity taken is small, or on a counterpoised filter, but we recommend the method of working which we employed for the estimation of stearic acid in mixtures of fatty acids (see ANALYST, vol. xxi., p. 322); but instead of filtering through cotton we find the best filtering material to be thin flexible chamois-leather tied over the end of the small thistle-funnel, from which any adhering precipitate can afterwards readily be removed by washing.

From 1 to 2 grammes of the glycerides are dissolved in 40 c.c. of ether, to which a few c.c. of glacial acetic acid are added, the precipitate forming being more granular from such a mixture than when ether alone is employed. The solution is cooled in an ice-chest and bromine added, the flask being preferably left all night in the ice. This, however, is not essential for ordinary working. The liquid is filtered off by the suction-funnel attached to a pump, the flask washed out with four successive portions of 10 c.c. of ether at 0° C., and the residue dried in the flask to constant weight. But even when ether at ordinary temperature is used, no considerable error is introduced.

Various samples of pure linseed oil were examined by this method, with the following results:

TABLE VI.

Sample.	Oil taken.	Weight of Precipitate.	Percentage of Deposit.
A	1·3226	0·3156	23·86
A	3·1005	0·7573	24·42
B	0·6792	0·1765	25·8
C	1·0000	0·2480	24·8
C	1·0000	0·2500	25·0

A sample of walnut oil gave, in two determinations, 1·9 and 1·42 per cent. of bromo-compound. Poppy oil (four specimens) gave no deposit, nor did brazil-nut oil, maize oil, cotton-seed oil, olive oil, Japanese wood oil, or almond oil. Mixtures of linseed oil and other oils gave percentages of bromine-compound in proportion to the percentage of linseed oil, as will be seen from the following table:

Oils Used.	Linseed Oil, Per Cent.	Insoluble Bromide, Per Cent.	Linseed Oil calculated from Bromide.
Linseed A and walnut ...	69	16·6	69
,, ,, ...	38·2	9·3	38·1
Linseed A and maize oil...	52	12·4	50·8
,, ,, ...	50·5	12·2	50·0
,, ,, ...	51·7	12·6	51·6

It will be seen from the above figures that the determination of the amount of the precipitate can usefully serve for testing the purity of unoxidized linseed oil. More extended investigation as to the variation in the proportion of the precipitate-yielding substance would, of course, be necessary, although as far as we have gone the variation appears to be small.

Considerable interest is attached to the nature and composition of the insoluble bromine-compound. From its origin it cannot be identical with the acid hexabromide; this is also shown by its melting-point, which is from 143·5° to 144° C., against hexabromide (177° C., Hazura; 180° C. to 181° C., Hehner and Mitchell). If it were hexabromo-linolenic glyceride, it would contain 62·28 per cent. of bromine. It cannot be linolic tetrabromo-glyceride with 52·23 per cent. bromine, since maize oil does not furnish any insoluble compound, while the free acids readily yield large amounts of the acid tetrabromo-derivative. We have made a considerable number of bromine determinations, which gave remarkably constant results. In various preparations we found 56·38, 55·7, 56·38, 56·32, 55·55, 56·17, and 56·32 per cent. bromine. This percentage is too low for the hexabromo-glyceride and too high for the tetrabromo-compound. Dr. Streatfield, of the Finsbury Technical College, was kind enough to make carbon and hydrogen determinations of a specimen of the material. Its ultimate composition was as follows:

Carbon	32·97
Hydrogen	5·42
Bromine	56·18
Oxygen	4·44
Ash	0·99
	100·00

Calculated for the ash-free material, the composition is as follows:

Carbon	33·29
Hydrogen	5·48
Bromine	56·74
Oxygen	4·49
	100·00

We are inclined to attribute to the bromo-compound the formula $C_{57}H_{96}O_6Br_{14}$, which would require:

Carbon	34·27
Hydrogen	4·81
Bromine	56·11
Oxygen	4·81
	100·00

It must be remembered that the substance, on account of its insolubility, could not be purified by crystallization. In addition, the presence of mineral matter, probably derived from the oil itself, would tend to make deductions from the results still more uncertain. The percentage of bromine in the substance strongly points towards a mixed bromo-ester, and we suggest, very tentatively, the following formula:

$$C_3H_5\begin{cases}C_{18}H_{29}O_2Br_6\\C_{18}H_{29}O_2Br_6\\C_{18}H_{33}O_2Br_2,\end{cases}$$

which, however, cannot be definitely accepted until a perfectly pure specimen has been examined. We are the more inclined towards a mixed glyceride formula, since the existence of such mixed esters has been fairly well proved in the case of butter-fat, and also because it is impossible to separate, even by persistently carried out recrystallization, the stearin from palmitin contained in animal fats. When, on the other hand, the glycerides are broken up by saponification, the separation is readily effected, and from the fatty acids separated from linseed oil a nearly pure hexa-bromide is readily obtained.

We have also made experiments with other classes of oils. Thus the acids of rape oil, which contains rapic acid, $C_{18}H_{34}O_3$ (which, unlike ricinoleic acid, does not appear to be a hydroxy-acid), yielded with bromine 3·6 per cent. of an insoluble bromo-acid containing 58·6 per cent. of bromine, and having a melting-point of 179° C. It was, therefore, neither the hexa- nor the tetrabromo-compound of Hazura. Rape oil itself gave 0·9 per cent. of insoluble precipitate only; this was not further examined. The similar mustard-husk oil gave a fine granular precipitate amounting to 1·5 per cent.

Some of the marine animal oils approach linseed oil in many of their properties, especially in their iodine absorption. It is interesting to note that the (impure) linolenic acid which we prepared from linseed oil had a marked odour of fish oil, and that pure linseed oil, when heated in steam, often emits a similar fishy smell.

The free acids prepared from cod-liver oil gave on bromination a white precipitate, which, when dried in the water-oven, became dark brown. It amounted to 18 per cent., and contained 62·91 per cent. of bromine (theory requires for the hexa-compound 63·31 per cent.). The air-dried substance had no definite melting-point, but decomposed below 200° C. Attempts were made to determine its melting-point in a sealed tube, but these also yielded no results.

The cod-liver oil itself (the glyceride) yielded with bromine an immediate precipitate, consisting partly of a solid substance, and partly of a heavy oil, which was difficult to separate from the former. Eventually a perfectly white solid substance was prepared, which amounted to 42·9 per cent. and contained 56·32 per cent. of bromine. It had no sharp melting-point, decomposition taking place before fusion occurred. Cod oil behaved in an analogous manner, yielding 35·5 per cent. of insoluble compound, which was not further examined.

Shark oil, similarly, gave 22 per cent. of a bromine-compound, and whale oil 25 per cent.

The investigations of Hazura, both as regards the hydroxy-derivatives and the

bromine compounds of the unsaturated fatty acids, are of such great importance that it seems surprising so little has been done by other observers since the publication of his papers. We trust that our small contribution to the subject will cause others to pursue research in this direction. We have already indicated in several places starting-points for further work, and we are convinced that valuable additions to our scanty knowledge of the unsaturated fatty acids would result. If our supposition of the existence of a compound glyceride of the unsaturated fatty acids be confirmed, it would probably furnish an explanation of the different properties of oils possessing similar chemical constants.

ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

FOODS AND DRUGS ANALYSIS.

Pumpkin-seed Oil and its Adulterants. H. Poda. (*Zeit. für Untersuch. der Nahr. und Genussmittel*, 1898, 625-628.)—This oil (*Oleum cucurbitæ*) is used in Austria and Hungary for dietetic purposes in place of olive oil, to which it comes next in price. Its usual adulterants are linseed oil, sesame oil, cotton-seed oil, and rape-seed oil. Such sophistication can be detected by comparing the iodine and refractometer numbers, the specific gravity and melting-points of the fatty acids. Two samples of the pure oil prepared by the author gave the following results:

	Description.	Iodine Number.	Saponification Number.	Refractometer Number at 25° C.
Oil I.	Cold pressed ...	124·49–123·91	189·31–189·51	70·2
	Hot pressed ...	123·69–122·85	189·24–189·37	71·4
Oil II.	Cold pressed ...	125·12–125·13	189·93–189·99	71·1
	Hot pressed ...	124·31–124·76	189·56–189·62	72·0

In the author's opinion pure samples should give results between the following limits:

Specific Gravity.	Iodine Number according to Hübl.	Saponification Number.	Melting-point of the Fatty Acids.		Refractometer Number at 25° C.
			Beginning.	Ending.	
0·923–0·925	122·76–130·68	188·36–190·17	26·5–28·5	28·4–29·8	70·0–72·5

H. H. B. S.

Methoxyl Determination applied to the Examination of Resins, Balsams, and other Drugs. G. Gregor. (*Oesterreich. Chem. Zeit.*, 1898, 253, 254, and 288-290.)—The author has recently published a modification of Zeissel's method for the determination of methoxyl (*Monatshefte für Chemie*, xix., 116; ANALYST, this vol., 297). In the present paper he gives the methyl numbers of certain resins, balsams, and drugs as furnished by his method. In the first of the following tables,

Bamberger's results are given side by side with those obtained by the author for the sake of comparison :

RESINS AND BALSAMS.

No.	Description of Resin or Balsam.	Methyl Number.			
		Gregor.		Bamberger	
1.	Aloë hepatica	4·2		3·9	
2.	Aloë lucida	0		0	
3.	Ammoniacum	8·6		11	
4.		9			
5.	Asafœtida	11·9		18	
6.		6·9			
7.	Gum benzoin, Siam	43·4	4·3	30	28·5
8.	Gum benzoin, Sumatra	25·5	25·3	16·5	16·2
9.		20·3			
10.		20·1			
11.	Benzoin (almond), Sumatra ...	20		13·3	
12.	Canada balsam	0		0	
13.	Copaiva balsam	0		0	
14.	Copal	0		0	
15.	Colophonium	0		0	
16.	Dammar	0		0	
17.	Dragon's blood	27·6		33·8	
18.		25·3		—	
19.	Euphorbium	0		0	
20.		2·8		0	
21.	Elemi	0			
22.		2·5			
23.	Pine resin	0		0	
24.	Galbanum	3·7		3·7	4
25.	Gamboge	0		0	
26.		2·4		—	
27.	Guaiacum	73·8	74·2	83·8	84
28.	Gurjun balsam ...	0		—	
29.	Jalap resin	0		0	
30.	Laudanum	0		0	
31.	Liquidambar styrax	4·5		0	
32.		3·6		—	
33.	Myrrh	13·5		13·6	13·2
34.	Gum mastic	0		0	
35.		1·9		—	
36.	Olibanum	6·4		5·3	
37.		16·7		14·4	
38.	Balsam of Peru	21·8	21·7		
39.		22·6			
40.	Scammonium	0		0	
41.	Sandarach	0		0	
42.	Venice turpentine	0		0	
43.	Balsam of tolu	41·6		46·8	
44.		41·7			

DRUGS.

No.	Description of Drug.	Methyl Number. Gregor.	Bamberger.
1.	Araroba	19·2	18·8
2.		22·2	
3.	Guarana	1	
4.		1	
5.	Lactucarium	1	
6.		1	
7.	Lupulin	1	
8.		1	
9.	Opium	19·4	19
10.		22·3	
11.		24	
12.	Foxglove leaves (Fol. digital. purp.)	4·5	
13.		4·9	
14.	Deadly nightshade leaves (Fol. belladonn.)	4·6	4·2
15.	Red Peruvian bark (Cort. Chin. rubr.)	8·8	8·3
16.	Calisaya bark (Cort. Chin. calis.)	14·2	
17.	Gray Peruvian bark (Cort. Chin. fusc.)	14·4	
18.	Cinnamon, Chinese (Cort. cinnam. Chin.)	11·5	
19.	Cinnamon, Ceylon (Cort. cinn. Ceyl.)	15·7	
20.	Ipecacuanha root	7·5	
21.		7·9	
22.	Rhubarb (Rdx. rhei Chin.)	5·2	5·5
23.		5·7	
24.	Senega root (Rdx. Senegæ)	14·7	
25.		15	

H. H. B. S.

TOXICOLOGICAL ANALYSIS.

Micro-Chemical Identification of Spermatic Fluid Stains. C. Kippenberger. (*Zeit. für Untersuch. der Nahr. und Genussmittel*, 1898, 602.)—The identification of traces of seminal fluid by means of the spermatozoa being not always possible, the author has investigated the method of Florence, depending upon the reaction with iodine and potassium iodide, with the view of its application to forensic work, and finds that this must be accepted with considerable reserve. The reagent is a solution of 1·65 grammes of potassium iodide and 2·54 grammes of iodine in 30 c.c. of distilled water. On mixing an aqueous extract of seminal fluid with this reagent, crystals of a dark-brown colour, varying in form from long-shaped rhombic tables to fine needles, are produced. The crystals vary somewhat in size, as well as in form, and are soluble in excess of the reagent, and in ether, alcohol, acids, and alkalies. The method of employing the test is as follows: An extract is first made by warming the stains or fluid with a little water on the water-bath. The extract is then acidified with hydrochloric acid, cooled, and dry ammonium sulphate added. which causes a separation to occur. The fluid is then filtered, a few drops of the filtrate mixed with the reagent and the mixture examined under the microscope.

The researches of Richter (*Wiener Klin. Wochenschrift*, 1897, 569) and of Lecco (*Wiener Klin. Wochenschrift*, 1897, 820) led to the supposition that the constituent giving rise to the reaction was cholin, either normally present in small quantities or produced by the decomposition of lecithin, a recognised constituent of the seminal fluid. The author's experiments do not confirm this, but lead rather to the conclusion that the reaction is due to xanthine bodies and to creatinine. Further, the author is not able to confirm the statement of Florence that the reaction is only obtained with human seminal fluid. Experiments with the seminal fluid of bulls, rams, gazelles, camels, foxes, asses, dogs, amphibious animals, and birds, all resulted in the characteristic reaction, though slight differences were observable in certain cases. Contrary to Posner's experience, the author finds that seminal fluid which has become putrid still gives the reaction, and this applies also to old stains.

With regard to other animal secretions, menstrual fluid, and, in one case, normal blood, gave a very similar reaction, which might be mistaken for the reaction with seminal fluid. Normal saliva and saliva from tuberculous persons gave negative results, as also did pus, unless mixed with blood. No reaction was obtained with urine, but, as this excretion contains small quantities of both xanthine bodies and creatinine, it is probable that the reaction would be obtained by the use of large quantities or of an extract. H. H. B. S.

ORGANIC ANALYSIS.

The Freezing-point of Glacial Acetic Acid. J. C. Umney. (*Pharm. Jour.*, 1898, 242.)—In the British Pharmacopœia of 1885 glacial acetic acid was described as containing "nearly" 99 per cent. by weight of hydrogen acetate. In the new Pharmacopœia (1898) the strength of the acid is given as 99 per cent., and it is provided that the acid shall have a specific gravity of 1·058, and that it shall remain solid until the temperature rises above 60° F. (15·5° C.).

The author points out that this solidification-temperature is not in agreement with the percentage. Rudorff (*Pharm. Jour.*, Series 3, ii., 241) states that the strength of glacial acid can be estimated with greater accuracy by determining the freezing-point than by titration with alkali, and gives the following table:

100 Parts of Mixture.			Solidifying Point. ° C.
Containing 0·0	of water	... ...	16·7
,, 0·497	,,	... ...	16·65
,, 0·99	,,	... ...	14·8
,, 1·477	,,	... ...	14·00
,, 1·961	,,	... ...	13·25

From this it is evident that glacial acetic acid containing actually 99 per cent. of hydrogen acetate does not remain solid above 14·8° C.

According to the U.S.A. Pharmacopœia, glacial acid contains 99 per cent. of absolute acid, solidifies somewhat below 15° C., and becomes liquid again at about 15° C.

The commercial importance of this apparently trivial point is shown by the fact that glacial acid is admitted into New Zealand at an *ad valorem* duty of 20 per cent.,

but if the acid falls below the British Pharmacopœia standard, 5d. per lb. duty has to be paid, or about five times as much as the proper duty. The New Zealand Customs have hitherto based their decision as to the strength of the acid on its liquefying-point after freezing, and regarding the statement of the British Pharmacopœia as absolutely accurate, they charge the 5d. per lb. duty on all acid melting below 15·5° C.

C. A. M.

Estimation of Formic Acid in Presence of Acetic Acid and Readily Oxidisable Organic Bodies. A. Leys. (*Ann. et Rev. Chim. Analyt.*, vol. iii. [8], pp. 255-260.)—The method is based on the reduction of mercuric acetate to the mercurous salt.

In the case of liquids containing acetic and formic acids, the total acidity is first determined in terms of the former. If the relative proportion of formic acid is very low, the liquid is diluted until the acidity measures 20 to 30 per cent.; but if the ratio is as 1 : 20 of acetic acid, dilution to 2 per cent. of acidity is necessary.

Ten c.c. are then taken, mixed with 20 to 30 c.c. of a 20 per cent. solution of mercuric acetate, and made up to 100 c.c. with water, the whole being heated to boiling-point within seven or eight minutes, and then immediately set aside to cool until the following day. The brilliant white crystals of mercurous acetate deposited are filtered through glass wool, brought on to the filter, then washed with 95 per cent. alcohol, acidified with 2 per cent. of glacial acetic acid (to prevent decomposition of any residual mercuric acetate), and finally with neutral 95 per cent. alcohol. After removing the alcohol by anhydrous ether, and evaporating the latter, preferably *in vacuo*, the precipitate is dissolved as rapidly as possible in nitric acid diluted with its own volume of water, and an aliquot part of the solution is precipitated with sodium chloride, the mercurous chloride being then weighed after drying at 100° C. The weight multiplied by the factor 0·0976 gives the corresponding amount of formic acid. Drying should not be prolonged more than is absolutely necessary, or a loss of weight, due to volatilization, will occur.

When the liquid under examination contains alcohol in addition to formic acid, 2 c.c. of glacial acetic acid must be added to the test sample before adding the mercuric acetate, to prevent precipitation of the latter. Should the proportion of alcohol be relatively high, the crystals of mercurous acetate formed may be granular and dense, instead of the fine scales obtained in the first-named case.

If both alcohol and acetic acid (the latter in sufficient amount) are present in the liquid, then no further addition of this acid is necessary.

The method is also applicable in presence of acetic and formic aldehyde, and is recommended by the author for the estimation of formic acid in natural spirits and commercial formaldehyde, as well as for the titration of formaldehyde after oxidation.

C. S.

A Very Sensitive Test for Bromine in Urine. A. Jolles. (*Zeit. anal. Chem.*, 1898, xxxvii., 439-440.)—This test is based on the fact observed by the author that, on treating a hot acid solution of urine with permanganate, any bromine compounds

present are decomposed with the liberation of free bromine, which can be readily identified by means of *p*-dimethyl-phenyl-diamine paper. This is prepared by dipping filter-paper into a solution (0·5 gramme in 500 c.c.) of the hydrochloride of *p*-dimethyl-phenyl-diamine, and leaving it to dry. This substance gives with bromine a red colouring matter, the probable composition of which is $NH : C_6H_4 : N(CH_3)_2Br$, and which, in the solid condition, has a metallic, green glittering appearance. The reagent-paper, when brought into contact with bromine vapour, gives a violet coloration in the centre, while the edges are blue, gradually changing into grey, and finally into brown. The reaction is extremely sensitive, and is capable of detecting 0·001 gramme of sodium bromide in 100 c.c. of urine.

The corresponding iodine reaction is much weaker, and the coloration of the paper (yellowish brown) is quite distinct, so that traces of bromine can readily be detected in the presence of large quantities of iodine. Chlorine, too, gives a very feeble reaction, which does not interfere with the bromine reaction.

The test is made in the following manner: 10 c.c. of the urine are acidified with sulphuric acid, and an excess of potassium permanganate is added. The flask is warmed on the water-bath, and a moistened strip of the reagent-paper placed in the neck of the flask.

The fluorescein-paper recommended by Baubigny (ANALYST, this volume, 23) is also very suitable for the detection of bromine liberated from urine by means of permanganate. C. A. M.

On the Coagulation of Albumin. G. Halphen. (*Jour. Pharm. Chim.*, 1898, viii., 173-175.)—In order to assist the coagulation of albumin in solutions of pepsin and in urine, the author has made experiments on the addition of ammonium salts. He finds that salts of ammonium, such as the chloride and the citrate, accelerate the coagulation of albumin. A sufficient quantity of a 10 per cent. solution are added to the albuminous solution before boiling, and the precipitate collected on a weighed filter, washed, dried, and weighed. C. A. M.

The Detection of Pyramidon (Dimethyl-amido-antipyrine) in Urine. A. Jolles. (*Zeit. anal. Chem.*, 1898, xxxvii., 441-442.)—Pyramidon is an antipyrine-derivative which has recently been prepared and recommended by Filehne (*Berlin Klin. Wochens.*, 1896, 48). It gives a characteristic bluish-violet coloration with ferric chloride, which, like the analogous red colour given by antipyrine, rapidly disappears. A fugitive violet coloration is also produced by it on treatment with a nitrite and sulphuric acid. The test which the author has worked out for its detection in urine is based on the fact that it is oxidized by halogens with the production of a violet coloration. The blue colour yielded with an alcoholic solution of bromine soon fades, but that obtained with an alcoholic solution of iodine changes after some time to red. Hydrogen peroxide gives a blue colour on warming; the action of permanganate is too energetic, and a colourless compound results which no longer reacts with halogens. Weaker oxidizing reagents, such as arsenic acid and alkaline copper solution, do not give a blue coloration. The behaviour of pyramidon towards the halogens is to be

chiefly attributed to the presence of the dimethyl-amido group, which, as has often been observed, readily reacts with oxidizing agents.

Most of these reagents give no characteristic results with pyramidon in urine. Ferric chloride produces the violet colour, but less markedly than in aqueous solution. On the other hand, alcoholic iodine is very sensitive, and by testing the urine with a very dilute solution (10 per cent. iodine solution diluted with 9 volumes of water) there is formed on the surface of the liquid a sharp violet red ring, which on standing changes to reddish-brown. The author finds that when pyramidon is taken in doses of 0·2 gramme twice a day it is readily detected in the urine by means of this test.

C. A. M.

Detection of Carbon Monoxide in Confined Air. F. Jean. (*Ann. et Rev. Chim. Analyt.*, vol. iii. [8], pp. 260, 261.)—The Marmet permanganate reagent being liable to reduction by other agencies than carbon monoxide present in samples of air, the author prefers to draw the air through a tube containing cuprous chloride solution, which, in presence of the monoxide, deposits a characteristic red precipitate.

C. S.

The precipitate thus formed is according to Berthelot (*Ann. Chem. Pharm.*, xcviii., 392) probably $Cu_2Cl_2.CO.2H_2O$.—Ed.

The Volumetric Estimation of Certain Alkaloids as Higher Periodides. A. B. Prescott and H. M. Gordin. (*Jour. Amer. Chem. Soc.*, 1898, xx., 706-724.)—By adding a solution of iodine in potassium iodide to a solution of an alkaloidal salt, the lowest periodide (in most cases a tri-iodide) is generally formed; if the iodine be kept in excess the highest periodide is formed. Of these latter some are stable and can be recrystallized, while others readily decompose in solution into free iodine and lower periodides. Atropine enneaiodide belongs to the first class; strychnine and brucine heptaiodides to the second. The only periodide of morphine which the authors could obtain was the tetraiodide already prepared by Jörgensen. Aconitine yielded (probably) a tri-iodide and a heptaiodide.

The formulæ and iodine factors of the higher periodides so far determined are:

	Ratio of Alkaloid to 1 of Iodine.	Alkaloid Factor of 1 c.c. of $\frac{N}{10}$ Iodine Solution.
Atropine, $C_{17}H_{23}NO_3.HI.I_8$ ...	0·2849 ...	0·0036048
Strychnine, $C_{21}H_{22}N_2O_2.HI.I_6$ ...	0·4390 ...	0·00555467
Brucine, $C_{23}H_{26}N_2O_4.HI.I_6$ ...	0·5179 ...	0·00655299
Morphine, $C_{17}H_{19}NO_3.HI.I_9$ ...	0·74918 ...	0·00947937
Caffeine, $C_8H_{10}N_4O_2.HI.I_4$ (Gomberg)	0·3834 ...	0·00485

The general method to be followed in estimating the strength of an aqueous solution of an alkaloidal salt is as follows: To about 10 c.c. of $\frac{N}{10}$ iodine solution, diluted with a little water, is added 1 c.c. of the acidulated alkaloidal solution, and the mixture well shaken for a few minutes. If the clear supernatant liquid does not retain a dark-red colour after the separation of the precipitate, the alkaloidal solution is too strong and must be diluted. When the right dilution has been ascertained, the acidulated alkaloid solution is made up to definite volume, and 10 to 15 c.c. of it run from a burette into a graduated vessel containing 25 to 30 c.c.

of $\frac{N}{10}$ iodine solution, diluted with a little water. The mixture is made up to definite volume and shaken, until the supernatant liquid is clear and of a dark-red colour. If not, the determination must be recommenced with a larger excess of iodine, as an addition of more of the iodine solution to the same mixture is out of the question, since the lower periodides when once formed might not take up more iodine. With morphine, which only forms one periodide, this precaution is unnecessary. When perfectly clear, an aliquot portion of the liquid is filtered off and the excess of iodine determined with standard sodium thiosulphate. Thus the quantity of iodine multiplied by the "ratio of the alkaloid to 1 of iodine" gives the quantity of the alkaloid in question.

The authors state that they have obtained satisfactory results by this method, in the determination of the total alkaloids in nux vomica and belladonna after extracting them with various solvents. They also confirm the accuracy of Gomberg's method of determining caffeine (ANALYST, vol. xxi., 193). C. A. M.

Notes on Taka Diastase. W. E. Stone and H. E. Wright. (*Jour. Amer. Chem. Soc.*, 1898, xx., 639-648.)—The authors have made a series of experiments on the action of taka diastase (ANALYST, this volume, 108) on potato-starch as compared with that of malt diastase. They found that its action at the outset was more rapid, the blue colour of the starch with iodine changing almost immediately to reddish and violet tints, while the starch paste was very rapidly liquefied. On the other hand, the complete conversion of the starch into compounds which no longer gave colour reactions with iodine was effected much earlier by the malt diastase, this final result being scarcely attained by the taka diastase after several hours. Parallel experiments, in which the solutions were examined with the polarimeter at stated intervals, showed that the products of the action of taka diastase had invariably a lower specific rotation than those from malt diastase, which the authors attributed to the more rapid conversion into maltose.

These results being unfavourable to the use of taka diastase in place of malt diastase in the quantitative determination of starch, the authors made further experiments with different materials in which a starch estimation is commonly required. One gramme each of air-dried finely-ground wheat, maize, and potato were heated to the boiling-point during thirty minutes with 50 c.c. of water. After cooling to 60° C., 10 c.c. of malt infusion were added to each, and the temperature maintained at 60° C. A second series was prepared in the same way, to each of which was added 0·05 gramme of taka diastase, and a third series in which the quantity added was 0·1 gramme, the temperature being maintained at 40° C. After four and a half hours all were tested with iodine, and it was found that in those treated with malt infusion the whole of the starch had disappeared from the potato, while the wheat and maize still showed traces under the microscope. In those treated with taka diastase all showed an abundance of unchanged starch. After seven hours the samples treated with malt infusion were quite free from starch, but the taka diastase samples still contained a large quantity, and even after an additional twelve hours' treatment the starch reaction had not disappeared.

As a further test, duplicate quantitative determinations were made of the starch contained in a specimen of air-dried wheat, the solvent in the first instance being a freshly-prepared infusion of malt, and in the second a solution containing 0·05 gramme of taka diastase. In each case the sugar was determined by titration with Fehling's solution, and the results calculated to starch. The treatment with malt extract gave 55·46 per cent. of starch, while the determination by means of taka diastase gave 52·94 per cent.

From these experiments, the authors conclude that under the usual conditions of the analysis taka diastase is unsuitable for the quantitative determination of starch.

C. A. M.

The Quantitative Estimation of Proteids. H. Schjerning. (*Zeit., anal. Chem.*, 1898, xxxvii., 413-422.)—In this paper the author sums up the results of his previous communications, and embodies them in a complete method for the estimation of proteid substances in solution (*cf. Zeit. anal. Chem.*, xxxiii., 263; xxxiv., 135; xxxv., 285; and ANALYST, 1898, 105 and 185).

The reagents required are:

1. A solution of tin chloride prepared by dissolving 50 grammes of tin in a weighed flask containing a sufficient quantity of boiling concentrated hydrochloric acid and a little platinic chloride. The solution is evaporated down to about 130 grammes, made up to a litre, and filtered.
2. A solution of normal lead acetate containing about 10 per cent. of the salt, and 10 to 12 drops of 45 per cent. acetic acid in a litre.
3. A 5 per cent. solution of mercuric chloride.
4. Pure, dry ferric acetate.
5. Dilute acetic acid containing 15 c.c. of 45 per cent. acid in a litre.
6. A solution of pure uranium acetate (about 10 per cent.), free from ammonia.
7. Pure, crystallized magnesium sulphate.
8. A solution of ordinary sodium phosphate, containing 0·4 per cent. of the crystallized salt.
9. A solution of calcium chloride (about 10 per cent.).

The solution of the proteids is first diluted so that 10 c.c. contains a quantity of total nitrogen corresponding with about 5 c.c. of decinormal acid. Under some conditions the precipitations are readily made, but when the solution contains little or no ash, it is necessary to add mineral matter (solutions 8 and 9). In order to determine whether this addition is required, the following test is made: If the number of c.c. of the proteid solution, which correspond with about 10 c.c. of $\frac{N}{10}$ acid, do on boiling not completely precipitate the iron from a solution of 0·8 grammes of ferric acetate dissolved in 40 c.c. of dilute acetic acid (reagent 5) and 50 to 100 c.c. of water, the proteid solution must be regarded as poor in ash, and the precipitations by tin, lead, and iron must be made after the addition of mineral matter (reagents 8 and 9).

The Tin Chloride Precipitation.—About 5 c.c. of the tin chloride solution (reagent 1) are added to 25 c.c. of the proteid solution. After stirring well, the

beaker is covered with a glass and left for from six to twenty hours. The precipitate is then collected on a filter and washed with cold water. If the proteid solution is poor in ash, 10 c.c. of calcium chloride solution (reagent 9) are added before the tin chloride, and the precipitate washed with a cold 1 per cent. solution of calcium chloride.

The Lead Precipitation.—To 25 c.c. of the proteid solution are added a sufficient amount of the lead acetate solution (No. 2), the amount varying with different substances. Care must be taken that the excess of lead is not too great, or part of the precipitate may be redissolved. After adding the reagent, the liquid is boiled, and the precipitate collected and washed with cold water. If the proteid solution contains little ash, sodium phosphate solution (No. 8) is added in the proportion of about three volumes to each volume of the lead acetate solution used, before boiling. Since the lead precipitate is somewhat soluble in the precipitating reagent, a correction is necessary, which the author has determined experimentally to correspond with 0·15 c.c. of $\frac{N}{10}$ acid for each 100 c.c. of filtrate and washings.

The Mercuric Chloride Precipitation.—Five c.c. of the mercuric chloride solution (No. 3) are added to 25 c.c. of the proteid solution, the liquid allowed to stand from four to twenty hours at the ordinary temperature, the precipitate filtered off and washed with a cold 0·5 per cent. solution of mercuric chloride, and the nitrogen it contains determined by Kjeldahl's method.

The Iron Precipitation.—0·8 gramme of ferric acetate is dissolved in 40 c.c. of the dilute acetic acid (reagent 5), and 50 to 100 c.c. of water in a beaker, and the solution heated to boiling, with constant stirring. 20 c.c. of the proteid solution are then added, and the liquid again brought to the boiling point. The precipitate is filtered off and washed three or four times with boiling water. The filtrate should be quite clear, and if this is not the case, an addition of from 15 to 25 c.c. of the sodium phosphate solution should be made immediately after the second boiling, the liquid being in the meantime stirred and kept boiling. With a little practice the right amount of sodium phosphate can be estimated. 20 c.c. have no injurious effect if these directions are followed, and only in exceptional cases is it necessary to add greater quantities (at most 25 c.c.).

The Uranium Precipitation.—25 c.c. of the proteid solution are mixed with 20 to 25 c.c. of the uranium acetate solution, the liquid heated to the boiling-point with constant stirring, and allowed to stand for some hours, or until the following day, in a dark place. The precipitate is washed with a cold 1 to 2 per cent. solution of uranium acetate. The correction necessary for the solubility of the precipitate corresponds with 0·10 c.c. of $\frac{N}{10}$ acid for each 100 c.c. of filtrate and washings.

The Magnesium Sulphate Precipitation.—Five or six drops of 45 per cent. acetic acid are added to 20 c.c. of the proteid solution, and the beaker placed in a water bath, which is kept at a temperature of from 33° to 36° C. From 18 to 20 grammes of finely powdered magnesium sulphate ($MgSO_4 + 7H_2O$) are added, with constant stirring, and the liquid allowed to stand for thirty minutes to one hour at the ordinary temperature, a stir being given from time to time. The precipitate is filtered off and washed with a cold saturated solution of magnesium sulphate containing 4 to 5 grammes of 45 per cent. acetic acid per litre.

The relation of the several precipitates is given in the following scheme:

Precipitate.		Contains the Proteids.	Precipitate.	Contains the Proteids.
Tin Chloride = *a*		Albumin I		Albumin I
Lead Acetate / Mercuric Chloride	= *b*	Albumin I, Albumin II, Denucleïn	Uranium Acetate = *d*	Albumin II, Denucleïn, Propeptone, Peptone
Ferric Acetate = *c*		Albumin I, Albumin II, Denucleïn, Propeptone	Magnesium Sulphate	Albumin I, Albumin II, Propeptone

The quantity of the different groups of proteids can thus be readily determined, as was shown in a former communication. The proteids precipitated by lead acetate and mercuric chloride are identical, but as the mercury usually gives more satisfactory results than the lead, the latter need only be used in exceptional cases. The author concludes his paper with tables of the results obtained with solutions of various kinds of proteids which are very similar to those given in his former paper. C. A. M.

The Analytical Methods of distinguishing the Nitrogen of Proteids from that of Amides. J. W. Mallet. (Bulletin 54, United States Department of Agriculture.)—The author has investigated various methods which have been proposed for separating these two classes of bodies, such as dialysis; interaction with nitrous acid; treatment with permanganate in presence of free alkali or acid; with sodium hypobromite; behaviour with cupric hydroxide; precipitation by alcohol; the behaviour of several new or little-used reagents, such as phenol, trichlor-acetic acid, formaldehyde, etc.; of tannin; and also that of phosphotungstic acid. This last reagent proved in most cases the most reliable one, and when applied under proper conditions afforded satisfactory results, though in certain cases tannin was found to be the preferable reagent. Some of the special facts on which the application of phosphotungstic acid depends are new, and the particular use made of these points of behaviour has not been hitherto described. The assumptions of Stutzer, that the proteids and allied substances are precipitated by phosphotungstic acid, and that the amides are not precipitated, must be qualified. Thus peptones are incompletely precipitated, while flesh-bases, such as creatine, creatinine, etc., are fully precipitated. Some substances also of an amidic nature give precipitates which are almost insoluble in water, but the solubility of which is much increased by rise of temperature. It was found that the substances experimented with might be divided into the three following classes:

(*a*) Those which, even in pretty strong solutions, give no precipitate with phosphotungstic acid.

(*b*) Those which are precipitated in strong solutions, the precipitate redissolving with more or less ease on heating the liquid (or on treating the precipitate with hot water) and reappearing on cooling.

(*c*) Those which are precipitated, the precipitate not being sensibly soluble, and

the supernatant liquid remaining clear on being heated along with the precipitate and subsequently cooled.

Under the first head fall glycocine, alanine, leucine, asparagine, aspartic acid, tyrosine, and allantoin; under the second, glutamine, creatine, creatinine, hypoxanthine, carnine, and urea. The precipitate given by peptone becomes clotted on heating, and dissolves to a considerable extent, being reprecipitated on cooling. Under the third head are included egg-albumin, fibrin, casein, legumin, globulin, vitellin, myosin, syntonin, hæmoglobin, albumose, gelatin, and chondrin.

The following amidic substances yielded precipitates with phosphotungstic acid, which were found to be soluble in hot water in the following proportions: That from betaine, 1 part in 71 parts of water at 98·2° C.; creatine, 1 : 107 parts at 98·1°; creatinine, 1 : 222 at 97·9°; hypoxanthine, 1 : 98 at 97·6°; carnine, 1 : 132 at 98·4°. It seemed possible, by precipitating with phosphotungstic acid, and washing the precipitate with hot water, to separate the amidic substances from all proteid and proteid-like bodies, except the peptones. These last are, according to most authorities, completely precipitated by tannin; hence this reagent disposes of the one case unprovided for by phosphotungstic acid.

The precipitants employed were 5 and 10 per cent. solutions of phosphoduodecitungstic acid in 2·5 per cent. hydrochloric acid, and 5 and 10 per cent. aqueous solutions of tannic acid.

The method proposed is given below nearly in the author's own words. It is stated, for the sake of simplicity, first as applied to the analysis of meat, raw or cooked, and the variations required in the examination of other classes of food materials are given afterwards.

A carefully selected and accurately weighed sample is to be ground in a glazed porcelain mortar with as much sharp-edged siliceous sand, previously heated to redness with free exposure to air, or with as much hard glass in small, sharp splinters similarly ignited, as will suffice to thoroughly subdivide the tissue and reduce it to the condition of a smooth pulp. Of this pulp, very carefully mixed, so as to insure uniformity, two aliquot parts are to be taken. In one the total nitrogen is to be determined by the well-known Gunning-Kjeldahl process, using a rather large proportion of sulphuric acid, so that no previous drying of the sample is needed. The other part is to be digested with cold water, filtered on to a nitrogen-free filter, and the residue washed on the filter with water at the same low temperature as long as it gives up soluble matter in sensible amount. Cold water is used to avoid action on and extraction of the gelatinoids. Creatinine is quite easily dissolved, as is also sarcosine; creatine with tolerable facility. Xanthine, hypoxanthine (1 : 300), and carnine (1 : 312), are less soluble.

The filtrate is then to be slightly acidified with acetic acid, heated to about 90° C., and again filtered from any coagulum. A little more sand or pulverized glass may with advantage be stirred in before bringing it on to the filter the second time.

To this second filtrate is to be added an acidified solution of phosphotungstic acid as long as a precipitate continues to form, avoiding any very large excess of the reagent. With a moderate amount of sand or pulverized glass added, to prevent the formation of a dense clot, the liquid and precipitate are to be heated to about 90° C.,

filtered, and the precipitate washed thoroughly on the filter with water at about the same temperature.

Assuming now that nitrogen is present in the sample under examination only in the two forms of proteids and simpler amidic compounds, the three filters used and their contents are to be submitted to the Gunning-Kjeldahl process. By subtraction of the nitrogen found from the total nitrogen previously determined, the amount of this element present in the simpler amidic compounds will be obtained.

In cases involving the presence of ammonia or its salts, nitrates, or alkaloids, the nitrogen occurring in these forms must be deducted from the total nitrogen before recording the residue as nitrogen of the simpler amides and amido-acids. In like manner a separation of lecithin, when present, may be effected by the use of ether as a solvent, followed by a mixture of ether and alcohol, in order to secure a complete extraction. A determination of phosphorus is made the basis of a calculation of lecithin-nitrogen, and this is in turn subtracted from the total nitrogen found.

When peptones are present, these are to be precipitated by tannic acid from the solution which has been acidified with acetic acid and heated. After this has completely cooled down, and before adding phosphotungstic acid, the filter on which the tannic acid precipitate is collected and washed with cold water is, with its contents, to be submitted to the modified Kjeldahl process, and the nitrogen obtained reckoned as part of the proteid nitrogen.

The several filters and precipitates from which the proteid nitrogen is obtained may either be treated separately by the Kjeldahl process, or, preferably, may all be brought together and submitted to this process in a single operation. If the latter course be pursued, it will be well to introduce each filter with its contents as soon as washed into the strong sulphuric acid, so as to avoid any possible decomposition and loss of nitrogen as ammonia until all the filters have been brought together and the moist combustion process can be proceeded with.

When proteoses are present, it may be well to make a check determination of their amount by saturation of the aqueous solution (after acidification with acetic acid, heating and subsequent cooling) with zinc sulphate, determining nitrogen in the precipitate so formed by means of the Kjeldahl process.

When gelatinoids are present, as may be the case with soups, stews, and meat extracts, hot water may be used at once for solution or washing the original material, and this with the advantage of facilitating the extraction of the less soluble simpler amides and amido-acids. These are, as a rule, more easily dissolved in the presence of a little free acid; hence acidification at an early stage of the treatment is advantageous. In a case in which tyrosine might be present, as in some vegetable materials, and possibly among unabsorbed residua of food, the use of hot water and the presence of free acid would greatly increase the solubility of this substance.

In food of vegetable origin where much starch is present it will be better to avoid the use of hot water at first, so that the solution may not be loaded with viscid material, rendering filtration difficult.

In all cases in which the food material to be examined is already fluid from the presence of water—as, for instance, soup, milk, and the like—filtration will of course at once be resorted to, being almost always much facilitated by the addition of sand

or pulverized glass, and only such further quantity of water will be used as is required for washing the undissolved matter left upon the filter.

In the presence of fat in large quantity it may be well first to remove this, or most of it, by extraction with ether. The simpler amidic substances are, as a rule, insoluble in ether, but by way of precaution the ethereal solution of fats might be shaken up two or three times with acidified water, and the watery fluid evaporated and tested for nitrogen.

In regard to the method of reporting results, the most important point is the separate statement of the amount of nitrogen present in the form of proteids and their congeners, and in the form of the simpler amides and amidc-acids. But in attempting to calculate from the nitrogen found under these heads the actual amount of the proximate nitrogenous constituents of the food material examined, the difficulty arises as to what factor should be used for multiplying the nitrogen found in each case.

The author suggests the following factors :

For proteids and allied substances, multiply nitrogen found by the ordinary factor 6·25.

For flesh-bases and simpler amides of animal origin in food-materials, multiply by 3·05.

For simpler amides and amido-acids of vegetable origin in food materials, multiply by 5·15.

For mixed amidic constituents of unabsorbed solid residua in digestion experiments, multiply by 9·45.

As a matter of general practice, it is recommended that in all statements of the results the actual amounts of nitrogen obtained should be given, so that if, with further knowledge of the nature of the proximate nitrogenous constituents present, the factor used in calculation be subsequently changed, the original experimental work will still retain its value. W. J. S.

Quantitative Estimation of the Bitter Principles in Hops. C. J. Lintner. (*Zeits. für d. ges. Brauwesen*, vol. xxi. [31], pp. 407-410.) In order to overcome the inconveniences arising in the ordinary gravimetric method for the extraction and drying of the bitter constituents, the author recommends titration of the extract with decinormal alkali, using phenol-phthalein as indicator.

Ten grammes of hops are placed in a half litre flask, and extracted by means of 300 c.c. of petroleum spirit (B. P. 30° to 50° C.) for eight hours, under an efficient reflux condenser, the flask being immersed to a depth of 2 to 3 c.m. in water kept at about 50° C. When the operation is completed, the contents of the flask are cooled to 17·5° C., made up to 505 c.c. (=500 c.c. of liquid) with petroleum spirit, and filtered into a stoppered flask. This should be performed as quickly as possible, to prevent the extraction of γ-resin, which would falsify the results.

One hundred c.c. of the filtered extract are titrated with alcoholic (96 per cent. by volume alcohol) decinormal potassium hydroxide. To facilitate the admixture of the liquid with the reagent, 80 c.c. of 96 per cent. by volume alcohol are added, and

10 drops of phenol-phthalein (1 : 100) solution are used as indicator. The reagent is added until the yellow colour of the liquid shows a decided red tinge, the end point being easily distinguishable after a little practice. A blank titration must be made to ascertain the volume of alkali consumed in neutralizing the petroleum spirit and alcohol.

The resulting values multiplied by 0·4 express the content of bitter principle in terms of lupulinic acid. C. S.

INORGANIC ANALYSIS.

The Determination of Technically Available Molybdenum in Molybdenum Glance. H. Bornträger. (*Zeit. anal. Chem.*, 1898, xxxvii., 438.)—Most of the methods employed for the determination of molybdenum give the total amount of the metal, and not that available for technical purposes, which, however, is most frequently required in a valuation of an ore. In the following method described by the author only the readily available molybdenum is estimated:

About 1 gramme of the molybdenum glance is digested in an Erlenmeyer flask for about two hours with 25 c.c. of strong nitric acid, until all the molybdenum has been converted into molybdic acid. Ammonia is then added to dissolve the molybdic acid and the liquid filtered. The residue is again digested with nitric acid and treated with ammonia, and the combined filtrates mixed with nitric acid and evaporated to dryness. The residue, consisting of molybdic acid and ammonium nitrate, is digested with dilute alcohol (50 per cent.), in which the ammonium nitrate is soluble. The molybdic acid left behind is collected on a weighed filter, washed with 50 per cent. alcohol, dried, and weighed; or preferably it is dissolved in 50 c.c. of normal ammonia, and the solution titrated with normal sulphuric acid, the difference being calculated into molybdic acid. C. A. M.

A New Volumetric Method for the Determination of Copper. R. K. Meade. (*Journ. Amer. Chem. Soc.*, 1898, xx., 610-613.)—The following method is claimed to be superior to the iodine and cyanide methods, both in rapidity and accuracy. The copper is brought into solution as sulphate, the greater part of the free acid neutralized with ammonia, the solution warmed, an excess of sulphurous acid added, followed by an excess of ammonium or potassium thiocyanate. The solution is filtered through asbestos with the aid of a filter pump, and the precipitate and filter, after being well washed, are heated in a beaker with a solution of caustic potash or soda. The reaction which takes place is shown in the equation

$$2CuSCN + 2KOH = Cu_2O + H_2O + 2KSCN.$$

The oxide is filtered off on asbestos, and washed with hot water. The precipitate and filter are again placed in the same beaker, and an excess of ferric chloride or ferric sulphate (free from nitric acid, free chlorine, or ferrous salts) added, together with a little dilute sulphuric acid.

The solution is then titrated with standard permanganate, and the iron equivalent

of the permanganate used, multiplied by 1·125 gives the weight of copper in the sample.

Instead of sulphurous acid, acid ammonium or sodium sulphite may be used to reduce the copper. Since copper is the only metal precipitated as thiocyanate from an acid solution, the presence of arsenic, antimony, bismuth, or zinc will not affect the results. The caustic alkali solution used to convert the cuprous thiocyanate into cuprous oxide must not be too strong, or some of the copper will go into solution. The author recommends a solution of about semi-normal strength.

The results obtained by this method in the analysis of a sample of pure copper were in close agreement with those of an electrolytic determination. The comparative figures given by the two methods with specimens of copper ore are shown in the subjoined table :

Sample.	Grammes.	Per Cent. Copper by Thiocyanate-permanganate Method.	Per Cent. Copper by Electrolytic Method.
1. Chalcopyrite I. ...	1·0	19·85	19·95
2. ,, ...	1·0	19·70	19·86
3. Chalcopyrite II. ...	2·0	6·16	6·37
4. ,, ...	2·0	6·21	6·30
5. Malachite	1·0	36·19	36·10
6. ,, ...	1·0	36·19	36·15
7. Tetrahedrite	1·0	21·05	21·06
8. ,, ...	1·0	20·90	21·18

The author states that after the ore has been brought into solution duplicate analyses can easily be made in from half to three-quarters of an hour. C. A. M.

The Determination of Uranium. **H. Borntträger.** (*Zeit. anal. Chem.*, 1898, xxxvii., 436, 437.)—A. Patera described a method of determining uranium which consisted in dissolving the ore in nitric acid, diluting with water, saturating the solution with sodium carbonate, boiling and filtering off, and washing the precipitate. The filtrate, which besides uranium contained only traces of other metals, was mixed with sodium hydroxide, and the precipitate washed, ignited, and weighed as $Na_2O.2UO_3$, corresponding with 88·3 per cent. of UO_3.

While this method gives good results with rich ores, it is otherwise with ores containing only 33 to 65 per cent. of UO_3, and especially with uranium sand, which contains only from 8 to 18 per cent. In such cases considerable quantities of silica are dissolved, and are weighed with the uranium. To obviate this the author recommends dissolving the ignited precipitate in hydrochloric acid, filtering off the silica, and again precipitating the uranium with sodium hydroxide or ammonia, and weighing it as $Na_2O.2UO_3$, or UO_3. The method is illustrated by the following results :

	I.	II.	III.	IV.	V.
Uranium ore taken, grammes	1·606	1·198	1·123	1·430	2·180
Crude sodium uranate, grammes ...	1·190	0·920	0·750	1·155	1·800
Containing SiO_2, grammes	0·026	0·040	0·025	0·037	0·040
UO_3 precipitated with NH_3, grammes	1·010	0·780	0·622	0·857	1·325
Per cent. of UO_3	62·90	65·70	55·40	59·90	61·00

C. A. M.

Determination of very small Quantities of Phosphoric Acid. A. Jolles and F. Neurath. (*Monatshefte für Chemie*, 1898, xix., 5.)—A solution of potassium molybdate is first prepared by dissolving 8 grammes in 50 c.c. of water, and adding 50 c.c. of pure nitric acid of 1·2 specific gravity. One hundred c.c. of this solution are required for 0·1 gramme P_2O_5, so that for any quantity below 0·001 gramme 1 c.c. is sufficient. Twenty c.c. of the solution to be tested, which must be clear and colourless, are placed in a test-glass, 1 c.c. of the molybdate solution added, and the whole warmed to about 80° C. The colour is then compared with that produced under exactly similar conditions by known quantities of sodium phosphate—*e.g.*, 0·001, 0·00075, 0·0005, etc. The following results show the degree of accuracy obtainable:

P_2O_5 taken, Gramme.	P_2O_5 found, Gramme.	Difference, Gramme.
0·001	0·001	0
0·00085	Between 0·001 and 0·00075 nearer to 0·00075 0·000812	0·000038
0·00061	Between 0·00075 and 0·0005 midway 0·000625	0·000015
0·0005	0·0005	0
0·0004	Between 0·0005 and 0·00025 nearer to 0·0005 0·000437	0·000037
0·0003	Between 0·0005 and 0·00025 nearer to 0·00025 0·000312	0·000012
0·00022	0·00025	0·00003
0·00016	Between 0·00025 and 0·0001 nearer to 0·0001 0·000138	0·000022
0·0001	0·0001	
0·00006	Between 0·000075 and 0·00005 0·0000625	0·0000025

Small quantities of inorganic salts, such as are found, for instance, in natural waters, have no influence upon the results. H. H. B. S.

Titration of Phosphoric Acid with Uranium. N. Orlow. (*Farmaz. J.*, 1898, xx., 283; through *Chem. Zeit. Rep.*, 1898, 233.)—As the standardization of

uranium solutions on pure sodium phosphate is troublesome, the author recommends the use of the double acetate of uranium and sodium—$Na(C_2H_3O_2).2UO_2(C_2H_3O_2)_2$—which forms anhydrous crystals, and can easily be obtained pure. F. H. L.

APPARATUS.

Lubricants for Glass Stopcocks. F. C. Phillips. (*Jour. Amer. Chem. Soc.*, 1898, xx., 678-681.)—With the object of producing a more suitable lubricant for glass stopcocks, the author has made a series of experiments with various substances. A mixture which gave very satisfactory results was: Pure unvulcanized rubber, 70 parts; spermaceti, 25 parts; and vaseline, 5 parts. The materials were thoroughly mixed while hot, the rubber being melted first and the others stirred in. Another preparation, which gave still better results, was made by mixing 70 parts of pure rubber with 30 parts of yellow unbleached beeswax. Old rubber does not answer well, as the mixture may become more or less granular and opaque. It is stated that this lubricant prevents glass taps from sticking, even when concentrated solutions of alkali are used, and is quite translucent in thin layers. C. A. M.

REVIEW.

COMMERCIAL ORGANIC ANALYSIS. By ALFRED H. ALLEN. Second edition. Vol. IV. London: J. and A. Churchill. Price 18s.

With Vol. iv. the second edition of Mr. Allen's treatise, the production of which has extended over a period of fourteen years, is brought to its long-hoped-for conclusion. We need say nothing as to the merits of the earlier portions of the work, since these have already been criticised in these pages and have stood the test of time and use. The concluding volume is worthy of its predecessors—*finis coronat opus*—and analysts are now placed in full possession of the greatest treatise on organic analysis which has ever appeared in England, or, indeed, in any country—a treatise which has become as indispensable in the laboratory as the classical work of Fresenius. Mr. Allen is to be congratulated on the completion of the work to which, as he himself tells us, he has devoted the best years of his life.

The volume under review commences with an account of the classification and the general properties and reactions of proteids or albuminous principles; a detailed description is then given of the proteids of the egg, blood-plasma, urine, and plants. The last section treats more especially of the composition of wheat, barley, and malt. We note that the author restricts the terms "proteid" and "albuminous" to the substances of which albumin is the type, whilst gelatin and kindred substances are classed under the head of proteoïds or albuminoïds. The word "album*en*" is also limited to its original signification—namely, white of egg—the term "album*in*" being applied to the most characteristic constituent of this and similar substances. These are welcome endeavours to secure uniformity in a nomenclature which is at present somewhat confused.

The succeeding 178 pages are devoted to the consideration of milk and milk-products; and this is, perhaps, that portion of the volume which will appeal more

especially to the public analyst. The average composition of milk, the methods for its analysis, the detection of adulteration, the question of standards and limits, the use of preservatives, etc., are discussed with ample detail, and the results of the most recent experiments are recorded. Cheese, cream, condensed milk, and other preparations of milk, are then dealt with in an equally comprehensive and satisfactory manner; butter has already been considered in vol. ii. We are glad to find that, through the courtesy of Dr. T. E. Thorpe, Mr. Allen has been enabled to include a description, taken verbatim from an as yet unpublished report to the Local Government Board, of the method now adopted in the Government laboratory for the analysis of altered milk. With the general principles of the method analysts are already acquainted, and it is now to be hoped that its value will be tested experimentally by independent observers. The analyses recorded in the report of samples of milk which have been kept for various periods—in some cases more than a year—certainly speak well for the process, which has, moreover, a scientific basis.

Meat and meat-products are next considered, and the composition and analysis of meat-extracts receives full attention. A bibliography of this latter subject, which has been so much in evidence of late years, adds to the value of this portion of the work.

Much of the following section, which deals with the proteids of digestion, has little to do with "commercial" analysis; but the author has judged it well—and rightly, as we think—to record all facts which he has found to be of practical or analytical interest. The most recent views of modern authorities on this difficult subject are here to be found, and an account of the ingenious speculations of Latham and others as to the constitution of albumin affords a relief to the more practical parts of the volume.

A chapter on hæmoglobin and allied substances concludes the discussion of the true proteids, and an excellent account of the chemico-legal examination of blood-stains is here given. We may remark that the author differs from most British authorities on this subject in attaching considerable value to the measurement of the *average* size of the blood corpuscles in the differentiation of the blood of man and other animals, even in the case of old and dried stains.

The remaining portion of the work treats of the proteoïds or albuminoïds, which are classed under the four heads of Collagenes or Gelatoïds, Fibroïds, Chitinoïds, and Keratoïds. Among other matters of practical or commercial importance here dealt with may be mentioned the analysis of gelatin, the valuation of glue, and the examination of silk, wool, hair, and other fibres.

A long list of addenda comprises notes on original publications which appeared too late to admit of being incorporated in the work. The recent alleged synthesis of albumin or peptone finds a place here, and it is pertinently pointed out that a process not involving the use of sulphur in any form cannot possibly result in the formation of a true proteid.

Those analysts who have been unable to procure the earlier volumes of Mr. Allen's treatise, which have long been out of print, will be glad to learn that they are being revised by Dr. Henry Leffmann, of Philadelphia. Vol. i. has already been issued, and Vol. ii. will follow shortly. T. S. & N. L.

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ERRATA.

Page 145, four lines from bottom, *for* "one-eighth of a gramme" *read* "0·8 gramme."
Page 292, seventeenth line from bottom, and all through abstract, *for* "cresol" and "homocresol" *read* "creosol" and "homocreosol."
Page 302, eighth line, *for* "tin" *read* "zinc."

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THE ANALYST.

JANUARY, 1899.

PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS.

THE monthly meeting of the Society was held on Wednesday evening, December 7, in the Chemical Society's Rooms, Burlington House, the President (Dr. Bernard Dyer) occupying the chair.

The minutes of the previous meeting were read and confirmed.

The following gentlemen were proposed for election as members of the Society: Mr. Harry Broadbent, F.I.C., Analytical Chemist to Messrs. Goodall, Backhouse and Co., Leeds; Mr. William Thomas Burgess, F.I.C., chemist in charge of the laboratory of Sir Edward Frankland, K.C.B., F.R.S., Reigate; and Professor Thomas S. Goodwin, the Veterinary College, Glasgow.

The President read the following list of officers and members of Council for the ensuing year, as nominated by the existing Council for election at the annual meeting:

President.—W. W. Fisher, M.A.

Vice-Presidents (who have filled the office of President).—M. A. Adams, F.R.C.S.; A. H. Allen; Sir Charles A. Cameron, M.D., F.R.C.S.; A. Dupré, Ph.D., F.R.S.; Bernard Dyer, D.Sc.; Otto Hehner; Alfred Hill, M.D., F.R.S.E.; J. Muter, M.A., Ph.D., F.R.S.E.; Thomas Stevenson, M.D., F.R.C.P.

Vice-Presidents (who have not filled the office of President).—Charles E. Cassal; E. W. T. Jones; S. Rideal, D.Sc.

Hon. Treasurer.—E. W. Voelcker.

Hon. Secretaries.—E. J. Bevan, A. C. Chapman.

Other Members of Council.—Arthur R. Ling; C. A. Mitchell, B.A.; James Nimmo; Thomas A. Pooley, B.Sc.; R. T. Thomson; Rowland Williams.

The names of those members of the Council who do not retire this year are Bertram Blount; Sidney Harvey; C. G. Moor, M.A.; F. Wallis Stoddart; John White; and W. C. Young.

Mr. J. F. H. Gilbard and Mr. Martin Priest were elected members of the Society.

An illustrated lecture was delivered by Mr. A. H. ALLEN on "The Use of the Micro-Spectroscope, and the Methods of detecting Blood in Chemico-Legal Investigations."

AN AUTOMATIC BURETTE.

By H. Droop Richmond.

(*Read at the Meeting, June* 1, 1898.)

Some years ago it was the custom in my laboratory to measure off the sulphuric acid for the Leffmann-Beam process by means of a 100 c.c. burette, which was filled by means of a siphon with a tap to it; the acid ran in at the top of the burette, and the operator had to stand by the burette till the acid reached the zero mark, and then to turn off the tap.

To prevent the possibility of accidental overflow of the acid, it occurred to me to fill the burette from the bottom, using a three-way tap, and to cork up the top with a cork carrying a tube of diameter so small that the volume of the acid in the tube could be neglected (Fig. 1). A trial convinced me that the method was slow, owing to the time that sulphuric acid took to run out of the narrow tube, and a little consideration revealed the fact that the error was much larger than was at first glance apparent.

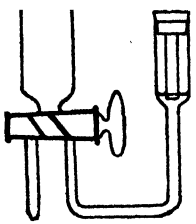


Fig. 1.

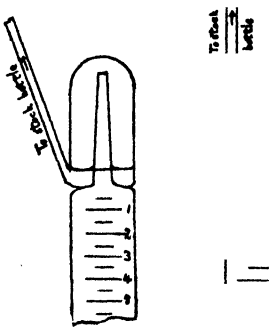


Fig. 2.

Fig. 3.

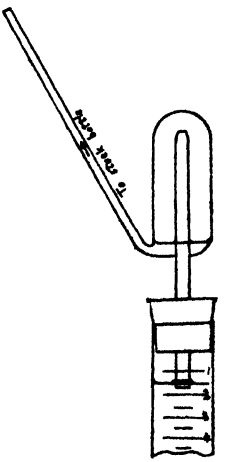
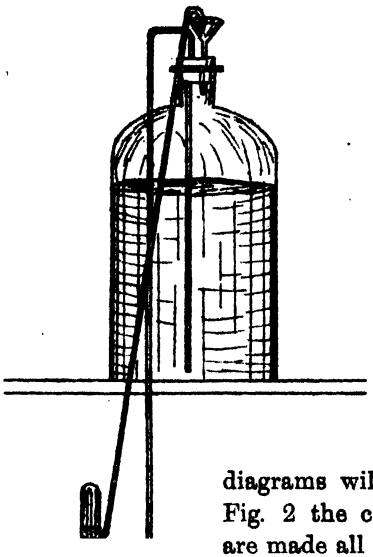


Fig. 4.

The following demonstration will show the nature of the errors introduced. The level of the acid a depends not only upon the level of the end of the tube, but on the pressure on the air in the space above; if h and h_1 represent the heights to which the acid rises when the stock-bottle is full and empty respectively, the volume of air in the space will vary in the proportion of

$$x \times \frac{760}{760 + ah \times \frac{d}{13{\cdot}6}} \quad \text{to } x \times \frac{760}{760 + a_1 h_1 \times \frac{d}{13{\cdot}6}}$$

where x = volume of air at 760 mm. pressure, d = density of liquid filling tube; taking x as 10 c.c., the distance from h to h_1 as 250 mm., and d as 1·82, which were the approximate values of the trial apparatus, a variation of nearly 0·5 c.c between the volume delivered when the stock-bottle is full and nearly empty is found; this variation is much greater than that due to the actual volume of acid in the tube, which was less than 0·2 c.c.; both variations go in the same direction, and the combined error quite unfits this apparatus for anything more than rough work.



A fairly obvious modification was to place the air-chamber above, instead of below, the effective zero point; this was accomplished by drawing out the end of the burette to a narrow tube, and enclosing this in an air-chamber. The following diagrams will show two forms of accomplishing this end. In Fig. 2 the chamber and an exit-tube for the excess of liquid are made all in one piece (*A* form); in Fig. 3 the exit-tube passes through a cork (*B* form). A further modification, which was not so accurate, but was convenient where great accuracy was not desired (*e.g.*, the measurement of sulphuric acid for the Leffmann-Beam process, for which I have used it for the last four years), is shown at Fig. 4 (*C* form). This enables an ordinary burette to be used, and, provided that the lower air-space is kept as small as possible, is quite accurate enough, and has the advantage that the zero point can be altered if desired, though it must be borne in mind that an alteration of the zero point will cause an alteration in the extent of the error. Of these forms *B* is preferable, and is capable of greater accuracy than *C*, and is not so liable to break as *A*.

In the case of corrosive liquids the connection between the stock-bottle and the burette is made by a siphon and the well-known air-trap, which enables an indiarubber connection to be used, and diminishes the risk of breakage.



Fig. 5.

The diagram (Fig. 5) shows a burette (*C* form) with all connections; after a measurement has been performed the tap is turned half round, and the burette is automatically filled to the zero point ready for use when required.

The liquid siphons over from the stock-bottle, fills the burette to the top, and then runs over into the air-chamber; from this it is forced up by the pressure of the incoming liquid up the ascending tube till

the level is equal to that in the stock-bottle; the zero is quite automatically adjusted and requires no attention.

When the liquid is run out, air enters through the ascending tube and bubbles through the liquid in the air-chamber. When the burette is again filled the liquid in the air-chamber is forced back into the bottle.

AUTOMATIC BURETTES.

By A. W. Stokes, F.I.C.

(*Read at the Meeting, November* 2, 1898.)

During the last few years I have interested myself in devising automatic apparatus for the purpose of rapidly, accurately, and safely measuring liquids, especially such liquids as strong ammonia and sulphuric acid.

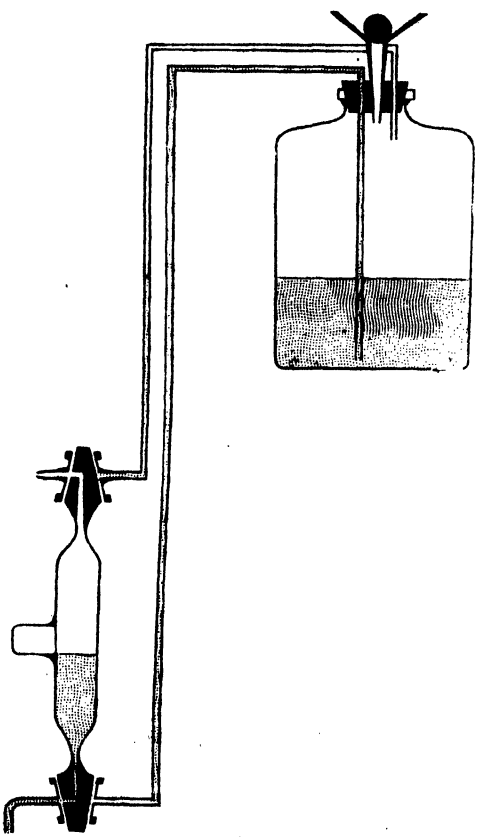


Fig. 1.

To be truly automatic, the apparatus should *without watching*—

1. Fill itself.
2. Empty itself.
3. Refill itself at the same time that it is emptying.
4. Be incapable of letting the whole of the supply run from the reservoir.
5. Need only one motion of the hand for all these operations.
6. Be adjustable to deliver any required quantity.

Only one of the apparatus I show you to-night fulfils all these requirements.

Fig. 1 is a plan I contrived that for exactitude excels all the apparatus hitherto introduced for this purpose. In this the measuring vessel, having a lateral opening at the top and another at the bottom, itself revolves. The lower end connected with the supply revolves in a sleeve, and the upper end, in communication with the overflow, revolves in another sleeve. The instant the measuring vessel is revolved all connection with the reservoir is cut off, so that an absolutely exact quantity is contained in the measure. Half a revolution brings its lower opening to the exit-tube, and its upper opening to an air-inlet. This form takes up its own supply of air, so

that the reservoir may be a closed vessel, if necessary. It, however, does not at the same time refill itself, nor can the quantity it delivers be varied without changing the measuring vessel.

Fig. 2 involves a new principle—that is, the introduction of a capillary tube into the measuring vessel so as to vary its capacity at will, and to avoid connecting the reservoir with any over-flow pipe. The capillary tube really forms an over-flow pipe, but as it is carried straight up above the level of the reservoir there can be no actual over-flow. By forcing this tube down into the measuring-vessel, the capacity of this latter is lessened, so that it will deliver a smaller quantity than before. The capillary tube also protects the cork through which it passes from being acted upon by any corrosive liquid that one may be measuring. But when the level of the liquid in the reservoir varies, it would apparently result that a varying quantity of liquid would be delivered, due to the greater or lesser height of the over-flow in the capillary tube. But a capillary tube may be used whose whole length would be filled by a single drop. Evidently, then, the variation of a few inches in the level of the reservoir will not vary the quantity delivered by more than some fraction of a drop.

Time, however, is lost with this apparatus, because it does not refill itself at the same time that it is emptying.

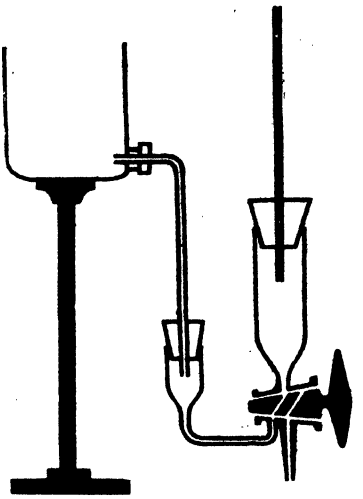


FIG. 2.

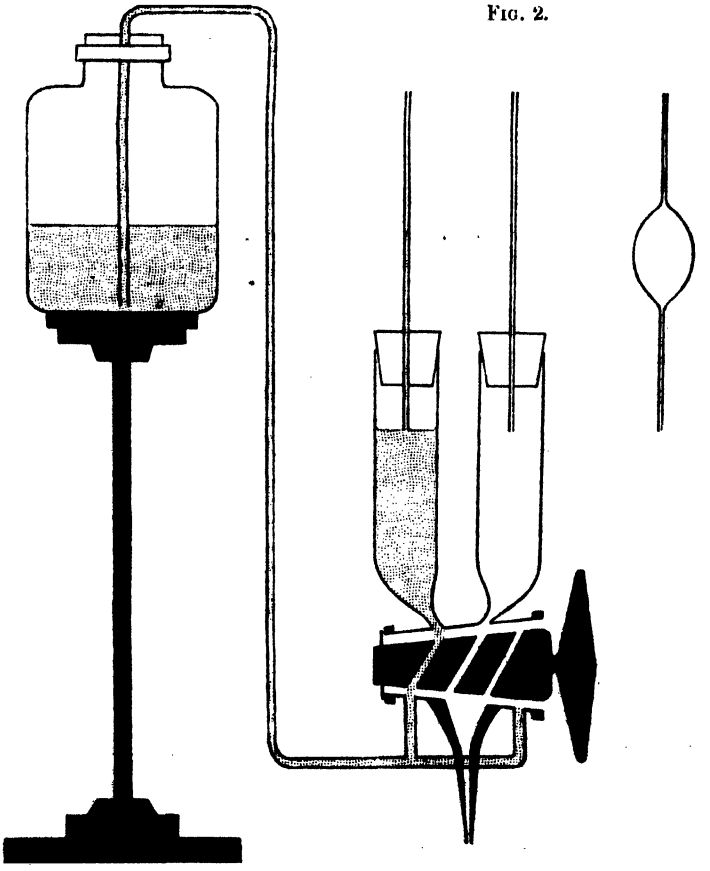


FIG. 3.

In Fig. 3 I duplicated the measuring-vessel, and used a capillary tube in each. By the kindness of Messrs. Lister, of Dursley, who have patented this and some other of my measuring devices, I am able to show you specimens to-night.

It fulfils all six of the conditions I noted at the beginning of this paper, as required of a truly automatic measure, and needs only a half-turn of the tap to repeat the measuring indefinitely. Its supply-pipe, acting as a syphon, can be dipped into any ordinary reagent-bottle without removing this from its shelf, and it will then deliver any required quantity.

When different quantities are required alternately, one capillary tube can be pushed down further than the other, so that one measuring-tube will, for instance, give 5 c.c., the other 10 c.c. at every half-turn of the tap. By using a capillary tube with a bulb blown on it accurate quantities far larger than the capacity of the measuring vessel can be delivered. The measuring vessel in such case serves only as a regulator, the bulb holding most of the quantity to be delivered.

ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

FOODS AND DRUGS ANALYSIS.

Alteration in the Character of Butter Produced by Feeding with Fat. G. Baumert and F. Falke. (*Zeit. für Untersuch. der Nahr. und Genussmittel*, 1898, 665-678.)—The authors give the results of a series of experiments undertaken with the view of testing the effect produced upon butter by feeding cows upon fats. The experiments were made upon two cows, Swiss and Dutch. The fats used—sesame oil, cocoanut oil, and almond oil—were selected on account of their dissimilarity from each other, as well as from butter itself. They were administered in the form of emulsions. The following plan of feeding, embracing five periods of twenty days each, was carried out: (1) Hay and rape meal only. (2) Hay and rape meal with sesame oil. (3) Hay and rape meal with cocoanut oil. (4) Hay and rape meal with almond oil. (5) Hay and rape meal only. The milk was passed through a separator and the cream churned to butter. The butter obtained was then at once melted at 40° to 50° C., allowed to stand for some time at the same temperature, and the fat clarified by filtration. Its examination included the determination of the Köttstorfer and Reichert-Meissl numbers, the Hübl and iodine numbers, the melting-points and refractions. The results showed that the butter fat was not only greatly changed in character through the feeding with the fats, but that the alteration, as indicated by the numbers obtained, was always in the direction of the numbers characterizing the three fats used in the experiments. In other words, the butter fats produced by feeding with sesame oil, cocoanut oil, and almond oil, exhibited the same characteristics as would be found in artificial mixtures of butter fat with the three oils in question. This is clearly shown in the following table of average results:

Description of the Periods.	Refraction.			Köttstorfer Number.			Reichert-Meissl Number.			Hübl Number.		
	Swiss Cow.	Fat administered.	Dutch Cow.	Swiss Cow.	Fat administered.	Dutch Cow.	Swiss Cow.	Fat administered.	Dutch Cow.	Swiss Cow.	Fat administered.	Dutch Cow.
Period I.	+1·7	—	+2·4	224	—	223	31·0	—	29·5	44·3	—	45·0
Sesame oil		+17·6			190			0·4			116	
Period II.	+5·6		+5·4	204		206	16·9		15·7	53·9		52·9
Cocoanut oil		−9·2			255			8			9	
Period III.	−0·5		−0·6	237		230	20·0		18·6	37·1		35·2
Almond oil		+13·6			195			0			98	
Period IV.	+3·6		+4·3	210		207	19·7		15·3	50·9		53·9
Period V....	+3·0	—	+3·6	218	—	216	22·0	—	24·4	41·2	—	44·5

The attempt to detect sesame oil in the butter produced in feeding with this oil by means of the furfurol and hydrochloric acid test was, however, not successful.

As regards the melting-point determinations, these yielded the following averages:

Period.	I.	II.	III.	IV.	V.
Swiss cow ...	34·16°	37·00°	33·58°	34·28°	34·50°
Dutch cow ...	33·96°	36·76°	34·76°	34·50°	35·15°

Compared with the melting-point of pure butter-fat, these results are all very high, the highest being in the case of the sesame oil feeding (II.), which agrees with the results of previous observers (*cf.* Reprint, vol. xxiii., p. 255). H. H. B. S.

On the Volatile and Insoluble Fatty Acids in Butter. R. Henriques. (*Chem. Rev. Fett u. Harz-Ind.*, vol. v. [9], pp. 169-172.)—The author investigated several samples of pure butter exhibiting unusually low Reichert-Meissl values, with the idea of devising a method for distinguishing these butters from mixed butters. It is based on the differences in the actual quantity of and the average molecular weight of the volatile and non-volatile fatty acids. The results, however, showed that butters with a normal R.-M. number contained 5 to 6 per cent., those with an abnormal 4 to 5 per cent. of volatile fatty acids, the average molecular weight of which varied from 93·3 to 99·8; hence the results have but a negative value for analytical practice.

With regard to the insoluble fatty acids, in these the Hehner number ranged between 86·5 and 89·1 for normal and 88·96 to 90·68 for abnormal butters; but a difficulty arose in the estimation of the average molecular weight, in that the values found on drying at 105° to 110° C. (viz., 267 to 270) differed from those (256·3 to 263) obtained when the drying was effected at the ordinary temperature in a vacuum desiccator.

The author, however, considers that, assuming butter to contain a large proportion of oleic acid, the above figures indicate that a considerable amount of an acid of lower molecular weight than palmitic acid is present; and that by isolating this acid and separating the unsaturated from the saturated acids it is not improbable, provided the assumption of a low average molecular weight for this undetected acid be found correct, that the presence of extraneous fats in butter may be discovered by the relatively higher molecular weight of the saturated acids they contain. C. S.

The Action of Formaldehyde on Flesh. E. Ehrlich. (*Zeit. Fleisch u. Milch. Hyg.*, 1898, viii., 232.)—The author has made experiments on the preservation of flesh by means of an 8 per cent. solution of formaldehyde. He finds that beef is preserved without the production of any unpleasant odour, but that the meat is only eatable for a short time after the treatment with the antiseptic. Horseflesh, on the other hand, invariably develops a characteristic intense smell within forty-eight hours, resembling that of roast goose-flesh. Only in one instance was a faint suspicion of this odour met with in the case of beef, and the author suggests that this difference may give a further means of distinguishing between the two kinds of flesh. C. A. M.

The Amount of Zinc contained in German Dried Fruit. P. Kulisch. (*Zeit. angew. Chem.*, 1898, 1015-1016.)—The author states that the zinc so frequently met with in American dried fruit is derived not from any addition of zinc salts, but from the action of the acid juices of the fruit on the zinc trays of the drying apparatus. On continued use, these become coated with a hard crust of dried juice, which largely prevents the fruit from coming in contact with the metal, and in such dried fruit the amount of zinc is naturally less. Since all modern German drying apparatus is constructed on the American plan, with zinc-wire trays, the author has examined different kinds of dried German fruit in order to determine to what extent they were contaminated with the metal. The quantities found in 100 grammes of fruit containing the usual commercial percentage of water were :

	Gramme.
Apple rings *a*	0·031
,, ,, *b*	0·021
Apple chips *a*	0·023
,, ,, *b*	0·027
Pear slices *a*	0·020
,, ,, *b*	0·026

All the samples thus contained an amount of zinc not much less than the mean quantity found in American apple rings (*cf.* ANALYST, xx., 251). C. A. M.

A Volumetric Assay of Opium. A. B. Prescott and H. M. Gordin. (*Jour. Amer. Chem. Soc.*, 1898, xx., 724-728.)—This is an application of the authors' general method for the estimation of alkaloids (*cf.* ANALYST, xxiii., 324). The opium alkaloids are liberated by the action of ammonia in the presence of certain solvents. The free narcotine, papaverine, codeine, and thebaine are removed by percolation with benzene, after which the morphine is extracted by percolation with acetone. The acetone is evaporated, and the residue taken up with lime-water, which completely dissolves and purifies the morphine. The solution is filtered, acidulated with hydrochloric acid, and the alkaloid estimated as periodide by titration with Wagner's reagent.

The details of the assay are as follows: 1 gramme of the finely-powdered opium is triturated in a mortar with 2 or 3 c.c. of a mixture of 5 c.c. of ammonia, 5 c.c. of alcohol, 20 c.c. of ether, and 10 c.c. of chloroform. The covered mortar is left for about three hours, with an occasional gentle shake, after which about 15 grammes of dry finely-powdered common salt are stirred into the pasty mass. It is then left in a warm place (30° to 35° C.) for two or three hours, and afterwards placed in a

desiccator containing sulphuric acid and a dish of paraffin wax until perfectly dry. The contents are next transferred to a small glass percolator (22 cm. by 1·3 cm.), and percolated with benzene until the percolate is colourless and gives no turbidity with Wagner's reagent. The dish beneath the percolator is replaced, and the percolation continued to complete exhaustion with acetone, as much as 200 c.c. of the solvent being usually required. The acetone extract is evaporated to dryness at a temperature not exceeding 45° C., the residue triturated with fresh lime-water, the solution poured without filtering into a stoppered cylinder of 100 c.c., the dish washed out with lime-water, and the liquid in the cylinder made up to 100 c.c. with lime-water. The cylinder is well shaken for half an hour, and filtered into a second cylinder until the filtrate amounts to 50 c.c. This liquid is rendered just acid with 10 per cent. hydrochloric acid and 25 c.c. of the $\frac{N}{10}$ iodine solution run in from a burette with constant shaking of the cylinder. After the addition of water to any convenient mark in the cylinder, the latter is shaken for about twenty minutes, until on standing the supernatant liquid (in which there should be an excess of iodine) is perfectly clear. An aliquot portion (one half) is then filtered off and the excess of iodine titrated with standard thiosulphate. The amount of iodine consumed by the alkaloid multiplied by the factor 0·0094793 gives the percentage of morphine in 0·5 gramme of the opium.

The precautions most essential are: (1) That the opium residue be thoroughly dry before percolation; (2) that the acetone should not contain anything distilling below 54° C. or above 58° C.; and (3) that after the excess of iodine has been added the cylinder be continuously shaken until the supernatant liquid is quite clear.

The following were the results obtained by this method in the assay of three samples of commercial opium:

		Opium Taken. Grammes.		Iodine Consumed by 0·5 gramme.		Morphine Per cent.
A	1	1·0036		0·116644		17·41
	2	1·008		0·116732		17·37
	3	1·0022		0·116642		17·44
B	1	1·0033		0·08872		13·25
	2	1·0023		0·08872		13·20
	3	1·0026		0·08872		13·21
C	1	0·9969		0·096319		14·48
	2	1·0016		0·097219		14·55
	3	0·9988		0·094552		14·19

C. A. M.

Estimation of Hydrastine in "Extractum Hydrastis Fluidum." **N. Rusting.** (*Pharm. C. H.*, 1898, xxxix., 787; through *Chem. Zeit. Rep.*, 1898, 289.)—Ten grammes of the extract are weighed into a large flask, diluted with 20 c.c. of water, and boiled and agitated till the total weight is reduced below 20 grammes. After cooling, water is added to make the weight exactly 20 grammes, a little infusorial earth introduced, and the whole filtered. Ten grammes of the filtrate are shaken with 25 c.c. of spirit and 3 c.c. of 10 per cent. ammonia; after a few minutes 25 c.c. of petroleum ether are run in, the liquid is again agitated, and 2 grammes of tragacanth are added. Forty c.c. of the clear ethereal liquid (equal to 4 grammes of the extract) are evaporated down to about 15 c.c. over warm water, and the vessel is put

aside for several hours in a cool place. The supernatant liquor is then cautiously poured off, and the hydrastine crystals dried in the water-oven and weighed.

F. H. L.

Notes on Estimating Eucalyptol. L. F. Kebler. (*Amer. Journ. Pharm.*, 1898, lxx., 492-494.)—Each species of the eucalyptus genus (about 120) apparently yields a distinctively characteristic oil, of which the United States Pharmacopœia recognises that distilled from the fresh leaves of *E. globulus*, *E. oleosa*, and some other species.

With regard to the differentiation of the official oils from the others the author points out that the specific gravity and rotatory power may be valuable criteria, but that the oil can be so mixed as to make these factors of little service. The phellandrene test is also serviceable, but the chief desideratum is to know the amount of eucalyptol in the oil.

In 1894 L. R. Scammell took out a patent for the preparation of eucalyptol on a large scale (*Eng. Pat.* 14,138), which was based on the fact that on adding concentrated phosphoric acid (specific gravity 1·75 to 1·78) to the oil at the ordinary temperature (not exceeding 60° F.), a crystalline compound of eucalyptol phosphate separated out, which, after being purified by pressure, or centrifugal force, was decomposed with hot water.

Faulding (*Chemist and Druggist*, 1895, 310) examined this process as applied to the quantitative estimation of eucalyptol and found that it gave excellent results. Helbing (*Pharm. Record*, xxxiii., 26), however, came to the conclusion that it was valueless for quantitative work, and the author's results confirm this.

By a modification of the process he has succeeded in obtaining closely approximate results in the following manner: Eight grammes of the oil are weighed into a beaker, which is cooled in ice-water. Four c.c. of phosphoric acid (1·75) are added, the beaker again placed in ice-water, and, after cooling, the contents thoroughly mixed by means of a glass rod. The eucalyptol phosphate is removed, purified by heavy pressure in folds of filter-paper, and weighed. It is then decomposed with hot water, the liberated phosphoric acid titrated with standard alkali, and the eucalyptol obtained by difference. In some cases it is necessary to add more phosphoric acid, especially with pure eucalyptol.

The results obtained by this method with pure eucalyptol and a number of oils are given in the subjoined table, in which the direct percentage of eucalyptol was obtained by Scammell's process.

No.	Specific Gravity at 15° C.	Grammes of Solid Eucalyptol Phosphate.	Per cent. of Eucalyptol, by difference.	Per cent. of Eucalyptol, direct.
1	0·9350	14·78	103·75	62·14
2	0·9153	10·70	73·62	31·20
3	0·9116	8·10	54·75	23·17
4	0·8912	7·13	43·62	24·29
5	0·8955	3·50	23·37	—
6	0·8876	did not congeal	—	—
7	0·9081	,,	—	—
8	0·9424	9·9	73·84	49·23

Of these samples No. 1 was pure eucalyptol, 2 and 8 were special oils at a high price, and the remainder such as are commonly met with in the wholesale trade.

C. A. M.

TOXICOLOGICAL ANALYSIS.

Piutti's Reagent for Alkaloids (Iodine-derivative of Para-ethoxyphenylsuccinimide). G. Simoncelli and N. Scarpitti. (*Gazz. chim. Ital.*, 1898, xxviii. [2], 171; through *Chem. Zeit. Rep.*, 1898, 279.)—The authors have investigated several iodine reagents for alkaloids, including those of Bouchardat and Selmi, and they find the most delicate to be one of the substances recommended by Piutti—viz., the iodine derivative of *p*-ethoxyphenylsuccinimide. This is prepared by treating the imide with a solution of iodine and potassium iodide in acetic acid. F. H. L.

Detection of Acetylene in Cases of Poisoning therewith. D. Vitali. (*Boll. chim. farm.*, 1898, xxxvii., 449; through *Chem. Zeit. Rep.*, 1898, 247.)—The suspected blood is mixed with 5 or 8 per cent. of pure acetone, placed in a retort, and distilled into an empty condenser and a set of absorbing bulbs containing more acetone. The retort is heated on a water-bath, the condenser and bulbs kept cold in ice. The acetone passes over, carrying the acetylene with it, and the latter may be identified by its characteristic reactions. Hydrogen sulphide and phosphide, which often occur in the crude gas, also dissolve in the acetone, and can be sought for therein. F. H. L.

Detection of Free Nitric Acid in Toxicological Work. D. Vitali. (*Oesterreich. Chem. Zeit.*, 1898, 333.)—The organs to be examined are warmed on a water-bath, water being added if needful, and freshly precipitated barium carbonate is introduced, a little at a time, as long as it dissolves. The mixture is then evaporated to dryness, and the residue boiled three or four times with an equal volume of absolute alcohol. By this procedure the calcium and magnesium nitrates are brought into solution, whilst sodium nitrate, the calcium nitrate naturally present in the organism, and the barium nitrate formed by the action of the free nitric acid upon the barium carbonate, remain in the residue. The alcoholic solutions are then evaporated to dryness, the residue taken up with water and neutralized very carefully with solution of barium hydrate. The fluid is then again evaporated to dryness, and treated with absolute alcohol. The undissolved residue is taken up with water, the solution treated with a little lead acetate, to precipitate organic matter, and filtered. The filtrate is then treated with hydrogen sulphide, to remove the lead, filtered, and the clear filtrate concentrated by evaporation, and dried in an exsiccator. The residue is examined for crystals of barium nitrate, which can be recognised by their crystalline form and by the usual tests. In this way it is possible to determine those nitric acid

compounds with albuminous bodies (acidalbumins) which are soluble in alcohol. The insoluble residue from the first treatment with alcohol contains, as already mentioned, the sodium nitrate, the barium nitrate resulting from the treatment with barium carbonate, and the calcium nitrate which must be regarded as a normal constituent of the body. It is made slightly alkaline with sodium carbonate, by which the nitric acid of the acidalbumins insoluble in absolute alcohol is neutralized, and the barium nitrate resulting from the free nitric acid is converted into sodium nitrate, whilst the calcium and sodium nitrates remain unchanged. The solution is filtered, the filtrate evaporated to dryness, and the residue treated with boiling absolute alcohol, which takes up the calcium nitrate, whilst leaving the sodium nitrate undissolved. The insoluble portion is dissolved in water, treated with a slight excess of lead acetate, the solution filtered, and the lead separated by hydrogen sulphide. The clear colourless fluid which is thus obtained is concentrated by evaporation, and dried in an exsiccator. Crystals of sodium nitrate in this residue can then be detected by the usual tests.

Another method which the author has used successfully is as follows: The intestines are cut into small pieces, and treated with freshly precipitated strychnine. As strychnine nitrate has an acid reaction, the neutralization cannot be controlled by means of litmus paper. The mixture is dialyzed, the residue twice extracted with warm water, the filtered fluids evaporated to dryness, and the residue treated with boiling alcohol. The alcoholic solution is then filtered, evaporated to dryness, the residue taken up with water, decolorized by treatment with lead acetate, followed by hydrogen sulphide, and the filtrate concentrated. Strychnine nitrate, formed by the union of the strychnine with free nitric acid, if present, would here crystallize out, and be determinable in the usual way. H. H. B. S.

Destruction of Organic Substances in Chemico-Forensic Work. C. Kippenberger. (*Zeit. für Untersuch. der Nahr. und Genussmittel*, 1898, 683-687.)—Villiers a short time since (*Compt. Rend.*, 1897, 1457) advised the use of a mixture of nitric and hydrochloric acids, with the addition of a solution of a manganese salt, for the destruction of organic substances. He stated that with this mixture the liver, spleen, and lungs could be oxidized in a few minutes, and the muscles within an hour—much more quickly, in fact, than by the use of potassium chlorate and hydrochloric acid, as recommended by Fresenius and Von Babo. The author is unable to confirm this, but found, on the contrary, that in order to complete the action it became necessary to add potassium chlorate from time to time with continued heating. The experiments made with Villiers' method, however, led to the trial of a manganese salt in conjunction with potassium chlorate and hydrochloric acid, and in this case the author found a decided acceleration of oxidation to result from the use of manganese. The most suitable manganese compounds for the purpose are manganic dioxide and manganous chloride, of which the former has the advantage of being a stock reagent in every laboratory. The sulphate is less suitable, on account of the possibility of metals in the case of metallic poisoning (*e.g.*, lead, mercury, or silver) becoming

converted into insoluble sulphates, and so causing difficulties in the subsequent analysis. It is sufficient to add a little manganic dioxide to the substance mixed with hydrochloric acid, and then to follow up with potassium chlorate in the usual way.

H. H. B. S.

ORGANIC ANALYSIS.

Detection and Estimation of Methyl Alcohol in Ethyl Alcohol. Trillat. (*Ann. et Rev. Chim. Analyt.*, vol. iii. [9], pp. 294-300.) — The author finds that ethylal and methylal are constantly formed amongst the products of the oxidation of ethyl alcohol, and that these furnish, when combined with dimethylaniline, bases differing in properties.

The base obtained from ethyl alcohol corresponds to the formula $C_2H_4 : 2[C_6H_4N : (CH_3)_2]$, and when dissolved in dilute acetic acid and treated with lead dioxide yields a blue solution, changing immediately to green, afterwards to yellow, and becoming completely colourless when boiled.

On the other hand, the base from methyl alcohol ($CH_2 . [C_6H_4N : (CH_3)_2]$) gives a persistent blue coloration, which is rendered more intense by boiling, and enables 1 part of the base to be detected in 200,000 parts of solution.

The same phenomenon is developed when the two alcohols are mixed, but in order to secure oxidation when the proportion of methyl alcohol is small, it is necessary to dilute the mixture with 10 volumes of water calculated to absolute alcohol, and it is advisable to employ 1·5 part of potassium dichromate to each part of absolute alcohol present. Another essential preliminary to the reaction is the careful rectification of the dimethylaniline to remove impurities.

In applying the test, 20 c.c. of the sample of alcohol are added to 300 c.c. of water and 30 grammes of powdered potassium dichromate, then cooled and mixed with 100 grammes of 20 per cent. sulphuric acid. After the reaction has proceeded for one hour the liquid is distilled, the first 50 c.c. (rich in acetaldehyde) being rejected. The distillate is neutralized with sodium carbonate and made up to 400 c.c. One-fourth of this is heated on a water-bath for five hours at 65° to 70° C. in a small closed flask, with 2 c.c. of rectified dimethylaniline and 1 c.c. of 10 per cent. sulphuric acid, with occasional agitation.

After neutralizing with alkali and distilling off the excess of dimethylaniline, a portion of the liquid is acidified with acetic acid, then treated with a few drops of water containing lead dioxide in suspension, and boiled in order to develop the blue coloration indicative of methyl alcohol.

The coloration is compared in the usual manner with typical samples prepared beforehand, and is sufficiently decisive to detect 1 per cent. of methyl alcohol. When the latter exceeds 5 per cent., the base may be weighed direct. C. S.

The Estimation of Caramel. Fradiss. (*Bull. Ass. Chim.*, 1898, xvi., 280.)— The substance under examination is mixed with methylic alcohol, which is evaporated on the water bath. The residue is dried first *in vacuo*, and then thoroughly in a

current of dry air. It is then extracted for two hours under a reflux condenser with methylic alcohol (95 per cent.), which dissolves all the caramel. The filtered extract is mixed with an excess of amylic alcohol which precipitates the caramel, and the precipitate, after being dissolved and reprecipitated two or three times, is finally dried on a weighed filter at 90° C. Various samples of molasses examined by this method were found to contain from 1·10 to 2·50 per cent. of caramel. C. A. M.

The Characteristics of Curcas Oil. O. Klein. (*Zeit. angew. Chem.*, 1898, 1012-1015.)—The *Jatropha Curcas*, from the seeds of which this oil is obtained, grows to a height of 3 or 4 metres, and is cultivated in all the Portuguese colonies, but especially in the Cape Verde Islands, which supply about three-fourths of the total quantity shipped to Portugal. There are two factories where the oil is extracted in Lisbon, the finely-divided seeds being subjected to hot pressure. The oil is used for lubricating and lighting, and in the soap and candle manufactures, and the pressed residue fetches a high price for manure.

The physical and chemical constants of the oil extracted in different ways by the author and of a commercial specimen were :

	Cold Pressed.	Hot Pressed.	Cold Pressed.	Extracted with Ether.	Commercial. Hot Pressed.
Specific gravity	0·9210	0·9240	0·9199	0·9207	0·9208
Refractive index at 25° C. ...	1·4686	1·4689	1·4687	1·4687	1·4681
Iodine value	109·8	109·1	110·4	109·1	107·9
Saponification value ...	203·5	203·6	197·5	197·0	198·1
Free acids	4·96	4·65	0·83	0·57	0·68
Volatile acids	7·10	5·5	—		

The oil is of a yellowish-red colour, which varies in shade according to the degree of pressure and heat to which the seeds were subjected. It has a characteristic odour, which cannot be readily mistaken. The acetyl values of the hot-pressed and extracted oil were 34·7 and 51·7 respectively by Benedikt and Ulzer's original process. By Lewkowitsch's method (*Journ. Soc. Chem. Ind.*), the corresponding figures were 17·6 and 25·3. The melting-point of the fatty acids determined in a capillary tube was 29·5° to 30·5° C., and the solidifying point 25·75° to 26·5°. The oil is soluble in alcohol to the extent of 24·12 per cent. It is insoluble in cold glacial acetic acid, but readily dissolves in the hot acid. The viscosity in Engler's apparatus at 20° C. was 9·45, so that in this respect curcas oil comes between crude rape-seed oil and olive oil, the latter having a viscosity of 9·03 to 10·3 (Künkler). The phytosterin determined by Bömer's method was 0·58 per cent.

According to Hiepe (*Rep. d. anal. Chem.*, v., 226), curcas oil is frequently used to adulterate olive oil in Portugal, but the author states that he has never yet met with any instance of such adulteration, although he has examined hundreds of suspicious samples of olive oil. Moreover, he considers such an admixture extremely improbable, taking into account the facts that 10 drops of curcas oil have a greater purgative effect than a tablespoonful of castor oil, and that an addition of 10 per

cent. of curcas oil to olive oil imparts an unmistakable odour. For its detection in olive oil Hiepe recommends treating the oil with nitric acid and copper filings, the resulting elaidin being of a reddish-brown colour. The author confirms this, but shows that the reaction is not conclusive, since earth-nut and cotton-seed oils give a similar coloration. Other colour reactions are also inconclusive. In Becchi's test the commercial oil causes a slight reduction, but the fatty acids cause no reduction. In the author's opinion the best tests for curcas oil in olive oil are the characteristic odour, and the increase in the specific gravity, refractive index, and iodine value. The oil was found to contain 9·59 per cent. of solid fatty acids melting at 57·5° C., and with a molecular equivalent of 269·5, from which data they were judged to consist of 20 per cent. of stearic and 80 per cent. of palmatic acid. No myristic acid could be detected. The liquid fatty acids (80 per cent.), separated as lead salts with ether, yielded on oxidation by Hazura's method equal quantities of sativic and dihydroxy-stearic acid, from which it was concluded that they consisted of equal parts of oleic and linolic acids. No ricinoleic or linolenic acid was found.

C. A. M.

Oil of Paradise Nuts, Oil of Brazil Nuts, Maize Oil. G. de Negri. (*Chem. Zeit.*, 1898, xxii., 961, 976.)—Paradise nuts are the seeds of the *Lecythis sapucaia* (*zabucajo*), a large tree belonging to the *Myrtaceæ* found in Brazil and Guiana. The seeds are edible, having a pleasant taste resembling Brazil nuts. On extraction with petroleum ether, 50 to 51 per cent. of a clear, colourless, or faintly yellow oil is obtained, which quickly turns rancid, but not so fast as oil of Brazil nuts. The latter begins to solidify at 4° or 5°, and is quite solid at 0°; oil of paradise nuts yields a white deposit at 12°, and solidifies completely at 8°. It is soluble in ether and chloroform, insoluble in cold absolute alcohol, hardly soluble in hot water; it is insoluble in cold acetic acid, but dissolves in an equal volume of the hot acid.

When maize is ground the germs are usually removed, and the yield is about 1 per cent. They show the following composition: water, 11·55 per cent.; nitrogenous matter, 39·07 per cent.; fat, 12·5 per cent.; carbohydrates, 31·76 per cent.; mineral matter, 5·3 per cent. On extraction with petroleum spirit 8 per cent. of oil is obtained, which is clear, yellowish-brown, and possesses a characteristic odour. It solidifies at 15° to a yellow crystalline mass. It is soluble in ether, chloroform, and carbon disulphide, insoluble in cold absolute alcohol, soluble in 30 parts of hot spirit. At 65° it dissolves in an equal volume of glacial acetic acid. It saponifies slowly with alcoholic potash. Maize oil quickly becomes rancid; one sample kept for a year in a half-filled flask finally contained 43·86 per cent. of free (oleic) acid. According to their place of origin, the germs differ widely in composition; one specimen of oil remained liquid at 0° for several hours; the iodine and acid numbers also vary. Its colour reactions are: Haydereich, orange-yellow with violet spots; Brullé, black, afterwards blood-red, solid in twenty-four hours; Schneider, no colour; Becchi, very faint brown; Milliau, faint brown; Baudoin, no colour. On distilling maize, a residue containing 11 per cent. of solid matter and 5·5 per cent. of oil is recovered (Villon). Maize itself yields 0·5 per cent. of oil, rather darker in colour than that obtained from the embryos. The constants of these three oils are:

	Oil of Brazil Nuts.	Oil of Paradise Nuts.	Maize Oil.
Specific gravity	0·918	0·895	0·9245
Solidifying point	0°	4°	15°
Saponification number	193·36	173·63	182·81
Iodine number	106·22	71·64	115·17
Acid number (calculated as oleic).	1·433	3·19	5·65
Acetyl number	—	44·08	—
Melting point of fatty acids .	28°–30°	37·6°	39·5°
Solidifying point of fatty acids .	—	28·5°	29·7°
Iodine number of fatty acids .	106·04	72·33	123·27
Refractometer number at 15° (Zeiss-Wollny)	—	61·3–61·5	74·5

F. H. L.

[Oil of paradise nuts must not be confused with oil of grains of paradise. Grains of paradise, guinea grains, or Melegueta pepper (*Semina cardamomi majoris*) are the seeds of *Amomum melegueta*, an African plant of the ginger species. This oil is volatile (*cf.* ANALYST, 1898, xxiii., 12). Paradise nuts are also known as sapucaia nuts, or "monkey pots."—F. H. L.]

Henriques' Process for the Analysis of Indiarubber Articles. R. Thal. (*Chem. Zeit.*, 1898, xxii., 974.)—This process (*Chem. Zeit.*, 1892, xvi., 1644) consists, *inter alia*, in a separation of the true rubber from rubber substitutes by the action of boiling 8 per cent. alcoholic soda, filtering off the dissolved matter through paper to avoid contamination of the solution with particles of loosened loading material, etc. Thal observes that an error is introduced into the operation by the partial solubility of filter-paper in the reagent; and he finds that Schleicher and Schull's extracted papers "No. 589," previously dried to constant weight at 100° C., lose between 4·86 and 6·88 (mean 5·63) per cent. of their weight when 100 c.c. of 8 per cent. alcoholic soda and the necessary amount of water to remove alkalinity pass through them. Rubber goods which are not loaded and only lightly dusted with talc need not be actually filtered to separate the soluble from the insoluble portion; but loaded articles must be filtered for the reasons just mentioned. He suggests, therefore, that the "No. 589" papers 12·5 cm. in diameter be always employed, and that 5·5 per cent. of their weight be added to the extracted rubber residue as a correction for solubility; taking 4 grammes of the sample for analysis, this loss, if not allowed for, would lead to a deficit of about 1·42 per cent. in the residue calculated on the crude material.

F. H. L.

Vanillin Mixed with Acetanilide. R. Hefelmann. (*Apoth. Zeit.*, 1898, No. 49; through *Deutsche Chem. Zeit.*, 1898, xiii., 358.)—In this product (which has already been referred to, ANALYST, 1898, xxiii., 266) the acetanilide was detected by a nitrogen estimation. The adulterated vanillin began to melt at 61°, instead of between 79° and 82°. It was only partially soluble in dilute caustic soda, whereas it should have been easily and completely soluble. To isolate the impurity, an ethereal solution was repeatedly extracted with strong sodium bisulphite, when the vanillin passed entirely into the aqueous liquid in combination with the bisulphite; the ether was washed in a little water and evaporated. The residue exhibited the correct crystalline form, melted at 111° to 113°, yielded drops of aniline on warming with

concentrated potash, and gave a strong odour of isonitrile on addition of chloroform to the alkaline liquid. Acetic acid was also to be recognised by the ordinary tests.

F. H. L.

INORGANIC ANALYSIS.

The Assay of Telluride Gold Ores. C. H. Fulton. (*School of Mines Quarterly*, 1898, xix., 419-426.)—From the results of a series of experiments on the assay of gold ores containing tellurium, the author has come to the following conclusions: In order to obtain good results, it is essential to have a large excess of litharge, but in the case of rich telluride ores this should be obtained by reducing the amount of ore, rather than by increasing the litharge, since otherwise the lead button may be too large to cupel directly. In the crucible assay, with direct cupellation, which is considered the best, the fire should be moderately hot, and the length of time in the furnace should be from forty to fifty minutes. The assay should be conducted so as to give a button of good size (from 20 to 28 grammes). The cupellation should be made at a low heat with crystals of litharge forming on the cupel, and in accurate assays the crucible slag should be re-melted, the button cupelled, and the amount of gold added to the first assay.

Scorification of any kind leads to bad results with telluride ores, and the loss of gold on cupelling a large or brittle button directly is much less than if it were first rescorified. The amount of gold lost in the slag on scorifying directly is very great, amounting to as much as 5·6 per cent. in the assays described.

The loss of gold in a rich telluride ore is greater than in a low-grade ore, but the percentage of loss decreases with the richness of the ore.

These conclusions agree with the experience of F. C. Smith, who found that the loss of gold in the assay of ores containing tellurium was very great, if the charge was not prepared with the correct proportion of litharge. C. A. M.

Separation of Zinc, Copper, Mercury, or Bismuth from Aluminium. F. S. Havens. (*Zeits. anorg. Chem.*, 1898, xviii., 147.)—The process which the author has already described (ANALYST, xxii., 194, and xxiii., 109) for the precipitation of aluminium chloride in presence of ether and hydrochloric acid, is equally available for the removal of the other metals mentioned in the title. The residual zinc chloride is preferably converted into nitrate by repeated evaporations with nitric acid, then ignited in a platinum crucible and weighed as oxide. Copper is converted into sulphate, rather than nitrate, and also weighed as oxide.

The figures recorded by the author in two tables are highly satisfactory, showing maximum errors of −1·3 and +0·9 milligrammes in estimating about 0·5 gramme of alumina; −0·8 and +0·4 milligrammes (corrected) in the direct estimation of the accompanying zinc oxide (0·1 to 0·2 gramme); and −0·7 and +1·3 milligrammes in the case of copper oxide; while all the mean results are practically identical with those demanded by the theoretical composition of the several mixtures analysed.

F. H. L.

Analysis of Ferro-Tungsten. A. G. McKenna. (*Proc. Eng. Soc. Western Pennsylvania*, 1898, xiv., 171.)—0·5 gramme of the finely-powdered sample is mixed with 3 grammes of sodium peroxide in a copper* crucible, and the mass is raised for a minute to a dull-red heat, holding the crucible in tongs so as to agitate its contents and prevent the alloy from sinking to the bottom. The melt is extracted with water, a few c.c. of alcohol added to render manganese insoluble, and the whole filtered. The filtrate, containing the tungsten and part of the silica, is evaporated to dryness twice with hydrochloric acid, taken up in weak HCl, boiled, filtered, and the residue washed with 1 : 10 nitric acid (water would cause it to pass through the paper). The insoluble matter from the fusion, which contains the iron, manganese, copper, and some of the silica, is dissolved in hot dilute nitric acid and evaporated to dryness. It is dissolved in weak HCl, and the silica is collected on the same filter that already holds the tungsten, washing as before with nitric acid. The combined precipitate is ignited and weighed as $WO_3 + SiO_2$; then treated with 0·5 c.c. of sulphuric and 5 c.c. of hydrofluoric acid to volatilize the latter in the usual way. The iron and manganese are subsequently separated.

In the presence of chromium and aluminium, the filtrate from the tungsten should be precipitated with ammonia, the mixed oxides weighed together, dissolved in strong nitric acid and potassium chlorate, and the alumina thrown down alone by a small excess of ammonia. After adding nitric acid, the chromium in solution can be titrated with ferrous sulphate and permanganate, or reduced with peroxide and weighed as Cr_2O_3. Part of the aluminium will be found in the iron precipitate, and must be allowed for.

Carbon may be conveniently estimated by the same process, for it is entirely converted into sodium carbonate, which can be decomposed with acid, absorbing the gas in barium hydrate. Corrections must be made for the several impurities, carbonate, silica, and iron, in the sodium peroxide, for which reason a known amount must always be used. F. H. L.

Estimation of Molybdenum. H. Brearley. (*Chem. News*, 1898, lxxviii., 203.)—The author has investigated Chatard's gravimetric process, which depends on the precipitation of sodium molybdate with lead acetate; and also Schindler's volumetric method of titrating an acidified solution of a molybdate with lead acetate, using tannin as an external indicator. To the former, various limitations were ascribed by Fresenius and by Chatard himself; but the present experiments widen its range of usefulness and bring out the following points: It is not necessary to employ neutral solutions; some free acetic acid is advantageous, as it obviates any danger of precipitating lead if faint alkalinity has been overlooked. Two c.c. of 33 per cent. acid may safely be used when dealing with 0·1 gramme of molybdenum; but an excess tends to change the molybdate from a granular to a powdery nature, and

* The author finds that copper resists the action of sodium peroxide quite as well as nickel, while adventitious salts of the former metal are less troublesome in the course of an analysis.

renders it liable to pass through the filter. This may be overcome by washing with a mixture of 1 c.c. of lead acetate solution (7·896 grammes per litre) and 500 c.c. of water just acidified with acetic acid. It is not necessary to separate the precipitate from the paper before ignition; they can be burnt together, wet or dry, in a muffle. It is advisable, however, to carbonize the paper at the lowest possible temperature, because extreme heating to remove refractory carbon is disallowed by the instability of the lead molybdate. The precipitate is not reduced nor appreciably altered in weight by ignition in presence of carbon. It can be safely ignited at a moderate red heat; even at full redness, or the melting-point of sodium carbonate, it only loses about 1 per cent. in six hours.

The chlorides and nitrates of sodium and ammonium do not affect the process; ammonium acetate, as made by neutralizing 0·880 ammonia with 33 per cent. acid, in large amounts simply involves a second filtration of the precipitate. It does not appear to retain alkaline salts, and prolonged washing is therefore unnecessary. If the ignited precipitate is dissolved in hydrochloric acid, neutralized with ammonia, treated with excess of acetic acid and a little more lead acetate, the purified molybdate is never sensibly different in weight. Manganese, copper, cobalt, nickel, zinc, magnesium, and mercury (ic) exert no practical interference, and occasion no trouble beyond more careful washing or a reprecipitation of the ignited molybdate. Even the unpurified precipitate carries down with it but little of these metals; and their presence can generally be detected by a variation from the normal (creamy) colour of the product. Cobalt requires a second precipitation. Zinc molybdate is mostly dissolved by the usual excess of acetic acid; if only an opalescence remains, it may be neglected; if the liquid is distinctly cloudy, the free hydrochloric acid of the original molybdenum and zinc solution must be partly replaced by acetic. In presence of mercury, the liquid must be warmed or ammonium acetate added. Uranium contaminates the molybdate of lead more seriously; by one reprecipitation the error may be reduced to 0·5 per cent.; but repeated operations are needed to separate the last traces.

Schindler's volumetric process is quicker and gives equally correct results with pure alkaline molybdates; but as the end of the titration is approached, the colour eaction between the molybdate and the tannin becomes rather uncertain. The method may be made more accurate by employing the tannin indicator as long as it is available (generally this is to within 1 per cent. of the truth), then filtering off a little of the hot liquid and testing it with lead acetate and sodium molybdate; more of the necessary reagent is added to the bulk, and the test is repeated with the same filter until the titration is complete. The influence of other metallic salts has not been investigated.

The reaction between lead acetate and molybdenum is so delicate that in a solution faintly, but decidedly, acid with acetic acid, 1 part of molybdenum in 4 million produces a distinct cloudiness. F. H. L.

The Analysis of Commercial Chrome Yellow. Willenz. (*Bull. de l'Ass. belge.*, 1898, xii., 163-167.)—The author's objection to Wittstein's method of examining chrome yellow (*Dingl. polyt. Jour.*, ccx., 280) is that the lead chromate is not

completely decomposed by the digestion with sodium carbonate, and hence the residue cannot be correctly taken as barium sulphate. He has found, in fact, that the residue from pure lead chromate may amount to as much as 49 per cent.

The following method is recommended as giving satisfactory results. One gramme of the powdered material is digested with 100 c.c. of dilute hydrochloric acid (1·20) at a gentle heat, and the residue transferred to a filter and washed with hot water. The total calcium in the filtrate is precipitated as oxalate, the calcium present in the form of sulphate calculated from a determination of the sulphuric acid, and the calcium in the form of carbonate obtained by difference. The lead sulphate is determined by digesting the residue with 50 c.c. of a neutral or slightly alkaline solution of ammonium acetate (specific gravity, 1·04), filtering, and evaporating the filtrate to dryness with an excess of sulphuric acid.

After the addition of 50 c.c. of water, the residue is boiled for about ten minutes with 25 c.c. of a solution of potassium hydroxide, containing 112 grammes of KOH per litre. This converts the lead chromate into potassium plumbite, which dissolves, leaving behind the barium sulphate and clay.

A separate estimation of the amount of chromic acid is made by Bunsen's iodometric method. C. A. M.

The Analysis of Incandescence Mantles. E. Hintz and W. Weber. (*Zeit. anal. Chem.*, 1898, xxxvii., 94-111.)—The mantles which have come under the authors' notice during the last three years have been composed almost without exception of thoria (96·42 to 99·26 per cent.) and ceria (0·49 to 2·02 per cent.), whilst the other rare earths, neodymia, lanthana, yttria, and zirconia, have only been present in minute quantities. Lime (0·1 to 1·05) and magnesia (trace to 0·21) were almost invariably present, and in some cases traces of silica.

With the object of determining whether these minor constituents had any influence on the light-emitting properties of the mantles, one of the authors (Hintz) has made a series of photometric experiments, from which he has arrived at the following conclusions:

1. Zirconia, lanthana, and yttria added in small quantities up to 1 per cent. do not increase the illuminating capacity of pure thoria mantles, but, on the contrary, tend to lower it. A very small addition of neodymia is also without much effect, but when the amount reaches 1 per cent. there is a slight increase in the quantity of light emitted.

2. The addition of neodymia, lanthana, or yttria (up to 1 per cent.) to mantles composed of thoria (99 per cent.) and ceria (1 per cent.) does not increase their illuminating power. Similarly, an addition of from 0·2 to 1 per cent. of zirconia has no effect, but the influence of 0·1 per cent. is doubtful.

3. An addition of zirconia, neodymia, lanthana, or yttria (up to 1 per cent.) to thoria-ceria mantles containing 0·5 per cent. of lime is without effect.

The authors have examined Knorre's volumetric method of determining ceria (ANALYST, this vol., 191), and find that the results thus obtained are as reliable as those of the gravimetric method. For the volumetric estimation of ceria in the

quantity in which it occurs in mantles they proceed as follows: 100 c.c. of the solution containing about 1 gramme of thoria and 0·1 gramme of ceria are acidified with from 5 to 7·5 c.c. of dilute sulphuric acid (1 : 6), diluted to 200 c.c., and the cerium compounds oxidized into the ceric state by adding three small successive portions of ammonium persulphate in the cold, and then boiling for one or two minutes after the first two additions, and finally for from ten to fifteen minutes after the final addition (about 3 grammes of persulphate are required to oxidize 0·2 to 0·3 gramme of cerium). At the conclusion of the boiling an additional 2 c.c. of the dilute sulphuric acid is added. When completely cold a dilute solution of hydrogen peroxide is run in, and the liquid rapidly titrated with potassium permanganate.

The authors recommend the following simplified method for the analysis of unburnt mantles. A number of the stockings—not less than twelve—are weighed, and their upper and lower ends cut off, since the former are often dipped in a hardening solution, and the latter into a solution of a cerium salt, in order to make the total percentage of ceria appear higher. The middle portions are weighed and completely extracted with water containing a few drops of nitric acid. The fabric remaining is ignited, the ash fused with potassium bisulphate, the melt dissolved in water containing hydrochloric acid, ammonia added, the precipitate dissolved in nitric acid, and the solution added to the main solution, which is then made up to definite volume.

1. Neodymia is tested for spectroscopically in a portion of the concentrated solution.

2. An aliquot portion is treated with oxalic acid after removal of the free acid by evaporation.

a. The precipitate is collected on a toughened filter, washed, and tested for neodymia, lanthana, and yttria as follows: The precipitate is washed into a beaker, heated with a concentrated solution of ammonium oxalate, the liquid diluted, allowed to cool, and filtered after standing for a long time. As the authors have shown in a former communication (ANALYST, this vol., 81), the residue left after extraction with ammonium oxalate contains a little thorium oxalate, so that even in the absence of neodymia, yttria, and lanthana, the percentage of ceria obtained will be higher than the truth. The presence of lime has also an influence on the result. If, however, the amount calculated from the weight of the precipitate does not exceed that of the actual quantity of cerium present by more than 1 per cent., it may be at once concluded that neodymia, lanthana, and yttria are not present in sufficient quantity to affect the illuminating capacity.

If, on the other hand, the excess is more than 1 per cent., a further examination is required. The weighed precipitate is dissolved by heating with sulphuric acid in a platinum crucible and the solution tested for these earths.

b. The filtrate from *a* is evaporated to dryness, the residue gently ignited, dissolved in hydrochloric acid, the excess of acid removed by evaporation, and the zirconia tested for microchemically by evaporating a drop of the solution on the object-glass after the addition of a drop of a solution of potassium binoxalate. In the presence of as little as 0·1 per cent. of zirconia characteristic crystals of potassium zirconium oxalate are to be observed.

3. An aliquot portion of the solution after removal of the free acid is precipitated with oxalic acid, and the precipitate, consisting of the total rare earths excepting zirconia, ignited and weighed. Lime has an influence on the result, but for practical purposes the authors consider that it is not of great importance if, in a mantle containing, say, 98·4 per cent. of thoria and 1·2 per cent. of ceria, 0·4 per cent. of lime is calculated with the thoria (= 98·8 per cent.).

4. The ceria is determined volumetrically in another aliquot part of the solution. The result deducted from that obtained in (3) gives the amount of thoria, provided that not more than negligible quantities of neodymia, lanthana, and yttria are present. C. A. M.

Estimation of Carbon in Iron by Combustion. Rozycki. (*Monit. Scient.*, 1898, [4], xii., 636; through *Chem. Zeit. Rep.*, 1898, 254.)—2·3 grammes of steel, or 0·25 gramme of ferrochrome, in fine powder, are mixed with 20 grammes of pure alumina, placed in a boat inside a combustion-tube 60 cm. long, and ignited in a current of pure oxygen. The gas is led over red-hot copper oxide, and finally absorbed in baryta water. The baryta is decomposed with nitric acid in Wiborgh's apparatus, and the carbon dioxide measured. The combustion occupies thirty-five minutes with steel, 1½ hours with ferrochrome. F. H. L.

A Reaction of Metallic Sulphides soluble in Ammonium Sulphide. J. Ducommun. (*Schweiz. Woch. Pharm.*, 1898, 434; through *Deutsche Chem. Zeit.*, 1898, xiii., 346.)—When yellow ammonium sulphide is mixed with a small quantity of formaldehyde, the colour disappears, and after a short time a copious white precipitate is thrown down, which is entirely soluble in strong sulphuric acid. If the sulphide is first diluted till it is only faintly yellow, formaldehyde produces no precipitate, and the liquid remains perfectly clear. In the presence of arsenious sulphide, the solution is bleached by the formalin, while sulphuric acid yields a white precipitate insoluble in excess. Antimony gives an orange-red precipitate on addition of acid; stannous or stannic sulphide, a white; gold, a yellowish-white; platinum, a brownish-red; the latter metal also prevents the previous decolorization. The precipitates are evidently some organic compound of the respective metals, for they turn black on ignition. In all cases the liquids should be cold and highly dilute; and then the reaction is capable of detecting 2 milligrammes of arsenious acid, and somewhat more of either gold or platinum. Arsenic acid only behaves in a similar fashion after it has been reduced by boiling the ammonium sulphide.

The process may be employed in the course of ordinary qualitative analysis to avoid the use of sulphuretted hydrogen. The solution containing various metals is neutralized with ammonia, treated with ammonium sulphide, and boiled to dissolve platinum and reduce arsenic compounds. One part of the filtrate is diluted and mixed with formaldehyde and acid. If a precipitate is produced, the remainder of the filtrate is analysed in the regular way; but if no precipitate is formed, arsenic, antimony, and tin are absent, and the first sulphide precipitate, together with the mother liquor, is concentrated slightly, and boiled with strong hydrochloric acid in order to separate the mixture into a solution containing those metals whose sulphides

are acid-soluble and a residue of the heavy metals, such as copper, etc., which yield insoluble sulphides; these are then severally examined as may be necessary.

The reaction shows that many sulphides, usually considered to be insoluble in ammonium sulphide, are really a trifle soluble therein—viz., copper, iron, lead, mercury. It should also be useful for detecting arsenic in pharmaceutical preparations like bismuth subnitrate. F. H. L.

Estimation of Sulphides, Sulphites, and Thiosulphates. W. Feld. (*Chem. Industrie*, "17/1898"; through *Deutsche Chem. Zeit.*, 1898, xiii., 322.)—The fundamental reactions underlying the author's processes are as follow: (1) Sulphides of the alkalis and alkaline earths, whether dissolved or suspended in water, are decomposed by a strong solution of magnesium chloride, forming sulphydrate and hydroxide, which are broken up on boiling in a current of carbon dioxide into magnesium carbonate and sulphuretted hydrogen. The latter is absorbed in standard iodine. (2) Sulphites yield the theoretical amount of sulphurous acid on distillation with hydrochloric acid, and the gas is also absorbed by iodine. Thiosulphates cannot be estimated in this way, for on heating with hydrochloric acid they produce sulphuretted hydrogen and sulphurous acid, with the deposition of free sulphur. (3) But if a thiosulphate is treated with iodine, it is converted into tetrathionate, and the latter distilled with HCl in presence of aluminium is reduced to H_2S, which can be determined in standard iodine as before. Any sulphite present at the same time is oxidized at once to sulphate, and takes no further part in the operation. (4) Thiosulphates are decomposed by mercuric chloride, yielding mercuric sulphide and a sulphate. Sulphites are not attacked, so that they can be boiled with HCl, and the SO_2 absorbed as before. Excess of mercuric chloride is advantageous, and it may be employed in the solid state.

An Erlenmeyer flask, holding 300 to 350 c.c., is fitted with a rubber cork having two holes. Through one is introduced a stoppered tube-funnel reaching to the bottom of the vessel; its upper opening can be closed with a cork bearing a bent tube connected to a supply of pure carbon dioxide (conveniently a cylinder of liquid). Through the second hole of the main cork is passed a short tube joined to four Geissler's potash bulbs, the ends of the several pieces of apparatus being made to touch within the rubber connecting tubes. The last set of bulbs is joined to a 10-litre aspirator to maintain and regulate the current of gas, and to measure roughly the volume passed. The first bulbs are empty, serving as water-condensers; the second are charged with a sufficiency (5 to 40 c.c.) of decinormal iodine to absorb all the gas evolved; the third contain some more (2 to 15 c.c.), diluted if necessary with water; the fourth are filled with 5 or 10 c.c. of decinormal thiosulphate to catch any volatilized iodine. To analyse a mixture of sulphide, sulphite, and thiosulphate, the sample in solution or fine powder mixed with water is brought into the flask, the cock on the funnel closed, and the whole apparatus tested for leaks. If satisfactory, the funnel tube is raised out of the liquid, and about 1 litre of CO_2 is passed to drive out air (were this precaution omitted, part of the H_2S might be oxidized to free S, which, however, would be noticed in the empty bulbs). Twenty c.c. of a 25 per cent.

solution of magnesium chloride (specific gravity, 1·22) are run in through the funnel without admitting air, the tube is pushed down again to the bottom of the flask, the liquid is heated, and the gas turned on at such a speed that the aspirator is emptied in about forty-five minutes. The potash bulbs are then rinsed out and titrated, 1 c.c. of $\frac{N}{10}$ iodine being equal to 0·00895 gramme of BaS or to 0·0039 gramme of Na_2S. The bulbs are charged once more, mercuric chloride and hydrochloric acid added to the contents of the flask, and the whole distilled again; the amount of iodine consumed represents the sulphite, 1 c.c. being equal to 0·0063 gramme of Na_2SO_3. A fresh portion of the original sample is titrated with $\frac{N}{10}$ iodine till it turns blue (the quantity used need not be noted); the mixture is placed in the same flask, together with rolls of thin pure aluminium foil which partly project above the liquid, acid is added, the gas turned on, and finally the solution is gently warmed; this gives the thiosulphate; 1 c.c. of iodine is equal to 0·00395 gramme of $Na_2S_2O_3$. It is highly desirable to check the purity of the several reagents employed throughout the process, lest reducing gases be produced among them. F. H. L.

Direct Conversion of Potassium Iodide and Bromide into Chloride. F. W. Küster. (*Zeits. anorg. Chem.*, 1898, xviii., 77.)—Dry potassium iodide can be safely and quantitatively converted into chloride by ignition in a stream of chlorine at a moderate temperature, heating it (about 2·5 grammes) for half an hour in a porcelain crucible over a luminous gas-flame about 2 cm. high, the top of which is kept 3 cm. from the base of the vessel. Even if the temperature be raised for a time till the crucible begins to glow, the loss is very minute (0·3 milligramme), and it is doubtful whether this is actually caused by volatilization of chloride. Dry potassium bromide cannot be decomposed in this manner; but if the sample be moistened, a similar reaction takes place more slowly, and by the following modification becomes equally available for analytical purposes. 2·5 grammes of the bromide are brought into an Erlenmeyer flask 7 cm. in height, together with 1 c.c. of water and 1 drop of 10 per cent. hydrochloric acid; and a current of chlorine is admitted through the porcelain tube of a Rose's crucible. The flask is placed on a sheet of asbestos, with a second sheet 2 cm. beneath, on which plays a small pilot gas-flame. The temperature is raised till the bromine evaporates quickly without boiling. After an hour or an hour and a half, one of the asbestos shields is removed and the water driven off; then the heat is gradually increased until the remaining asbestos is visibly red. The whole operation is repeated once or twice to obtain constant weight; but if a smaller quantity of bromide is taken in the first instance, the reaction is often complete the first time. The result may be most simply calculated by the use of the formula

$$\%KBr = a + b\frac{g'}{g}$$

where $a = 267{\cdot}59$; $b = -267{\cdot}59$; g' the weight of the potassium chloride; g that of the original bromide.

The examples quoted by the author show yields of 100·00 and 100·01 per cent. when working on pure potassium iodide. A specimen of Kahlbaum's bromide gave

99·07 and 99·08 per cent. of KBr by the above method; 99·06 and 99·20 per cent. by ignition of the silver halide in chlorine (calculating from the potassium bromide itself); 99·16 and 99·10 per cent. by similar treatment (calculating from the silver compound).

Commercial iodide seldom contains any impurity except water; bromide contains nothing except moisture and chloride. F. H. L.

Volumetric Estimation of Combined Sulphuric Acid. M. Reuter. (*Chem. Zeit.*, 1898, xxii., 357.)—The author has submitted Andrews' method for the titration of sulphate solutions to a careful examination; and he finds, provided the operation is carried out exactly in the following manner, that its accuracy, speed, and simplicity leave nothing to be desired. 10 c.c. of the original solution, which should contain about 0·14 gramme of anhydrous sodium sulphate, are boiled with 150 c.c. of a solution of barium chromate prepared by dissolving 3 or 4 grammes of pure precipitated chromate in 1 litre of water by the aid of 30 c.c. of strong hydrochloric acid. The acid is neutralized with powdered chalk, and the precipitate filtered off and washed; the filtrate is thoroughly cooled, acidified with 5 c.c. (not more) of strong HCl, treated with 20 c.c. of a 10 per cent. solution of potassium iodide, allowed to rest for five minutes in a covered vessel and an atmosphere of carbon dioxide (to give time for the complete reduction of the chromic acid, yet to prevent oxidation of the HI), then diluted to 1 or 1½ litre, and finally titrated with decinormal thiosulphate. Three atoms of iodine correspond to one molecule of sulphuric anhydride. F. H. L.

Method of Preparing an Exactly Neutral Ammonium Citrate Solution. A. D. Cook. (*Jour. Amer. Chem. Soc.*, 1898, xx., 585-586.)—It has been stated by several chemists that an exactly neutral ammonium citrate solution for agricultural analysis may be obtained by allowing the solution to stand after ammonia has been added to the citric acid and the proper dilution made. The author, however, finds that this is only the case when the solution has a sufficient temperature to expel the excess of ammonia. By vigorous stirring sufficient heat for the purpose is caused by the chemical action, but if this is neglected the solution will be slightly alkaline. The method recommended for the preparation of the reagent is as follows: 740 grammes of commercial citric acid are mixed with 1,900 c.c. of 10 per cent. ammonium hydroxide. After vigorously stirring the liquid until the acid has all dissolved, the solution is made up to 4,000 c.c. with water. It is then again stirred and transferred to a large evaporating dish, where it is allowed to stand over-night. Finally, it is transferred to the reagent bottle, brought to 20° C., and water added until the specific gravity is 1·09. C. A. M.

Estimation of "Available" Phosphoric Acid in Thomas Slag. J. Freundlich. (*Chem. Zeit.*, 1898, xxii., 974.)—Wagner has lately shown (ANALYST, 1897, xxii., 334) that in slags containing much silica, this substance may be prevented from contaminating the precipitate of ammonium magnesium phosphate, if the freshly

prepared citrate extract be treated with magnesia mixture previously mixed with alkaline citrate solution. Many slags, however, also contain sulphide of iron and calcium; and these bodies, being decomposed by the citric acid, yield sulphuretted hydrogen, which does not entirely escape, but, combining with the ammonia afterwards added, precipitates the iron once more, producing a blackish phosphate that becomes red on ignition. To avoid this error, a second precipitation should be resorted to whenever the slags contain much sulphur; and the ferrous sulphide in the first precipitate should be oxidized with nitric acid or aqua regia. F. H. L.

Estimation of Perchlorate in Chili Saltpetre. O. Foerster. (*Chem. Zeit.*, 1898, xxii., 357.)—The ordinary method of reducing the chlorates and perchlorates in commercial sodium nitrate by heating the mass to a red heat has several disadvantages. If the temperature be too low, they are not completely decomposed; while on the other hand, chlorine is apt to be lost by volatilization of the sodium chloride. The author's process is very exact, and does not demand the use of any particular degree of heat. 10 grammes of the sample are mixed with an equal weight of dry sodium carbonate (free from chlorine), and the whole is heated in a covered platinum or capacious porcelain crucible over a large flame for about ten minutes, till the mass is in tranquil fusion and no more bubbles of gas are given off. The melt does not creep up the walls of the vessel, and it is readily soluble. It is finally dissolved in excess of nitric acid, and the total chlorine is determined as usual. F. H. L.

(*Cf.* Abstract, ANALYST, xix. 221.)

Estimation of Perchlorate in Saltpetres. N. Blattner and J. Brasseur. (*Chem. Zeit.*, 1898, xxii., 589.)—Five or ten grammes of the dried potassium or sodium nitrate are heated for fifteen minutes over a Bunsen flame with 8 or 15 grammes of calcium oxide, carbonate, or preferably hydroxide. The mass is dissolved in pure nitric acid, the total chlorine determined by any of the usual methods, and deducted from that already existing as chloride in the original sample. The process is more convenient than those in which alkali-metal carbonates are employed, because the mixture does not melt, and thus is more readily soluble, while there is no danger of loss by volatilization, etc.

In three different ship-loads of "refined" Chili saltpetre, supposed to contain at least 96 per cent. of sodium nitrate and less than 1 per cent. of sodium chloride, the authors discovered from 0·12 to 1·01 per cent. of NaCl, and from 0·42 to 0·77 per cent. of $NaClO_4$; while a sample of potassium nitrate, "pure for analysis," contained 0·47 per cent. of perchlorate, but no chloride. F. H. L.

(*Cf.* ANALYST, xix., 221.)

Microscopic Detection of Perchlorate in Chili Saltpetre. M. van Breukeleveen. (*Rec. trav. chim. des Pays-Bas*, 1898, xvii., 94; through *Chem. Zeit. Rep.*, 1898, 145.)—Behrens' test for a perchlorate, which depends on the recognition under the microscope of the rhombic and almost insoluble crystals of rubidium perchlorate, can only be used in the examination of sodium nitrate when the impurity (calculated

as potassium perchlorate) amounts to at least 0·6 per cent. To detect smaller quantities (0·2 to 0·6 per cent.) 10 grammes of the sample are dissolved in 10 c.c. of hot water, diluted with 50 c.c. of 95 per cent. alcohol, raised to the boiling-point, and allowed to cool for one or two hours. The clear liquid is then poured off, evaporated to dryness on the water-bath, the residue taken up in as little water as possible, and Behrens' test applied. If the rubidium salt is coloured with permanganate, only enough of the latter must be added to give the liquid a faint pink tint when it is placed on the microscope slide and held over a sheet of white paper; or crystals of rubidium permanganate may be mistaken for those of the perchlorate.

F. H. L.

A False Nitrous Acid Reaction in a Drinking Water. A. Bömer. (*Zeit. für Untersuch. der Nahr. und Genussmittel*, 1898, 401.)—The attention of the directors of a waterworks having been called to a strong nitrous acid reaction given by the water, the matter was referred to the author for report. The addition of zinc iodide and starch and sulphuric acid certainly produced a deep blue colour. On careful examination, however, the water was found to contain some suspended matter which proved to be manganic dioxide, the presence of which, in conjunction with the chlorides in the water and the added sulphuric acid, would be sufficient to account for the reaction. After separating the suspended matter from the water the reaction was not produced.

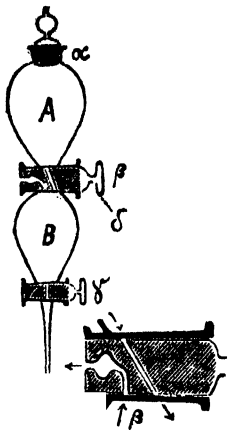
A short time afterwards the author received from the same company for examination a piece of lead pipe containing a brownish-black incrustation, which proved upon analysis to consist principally of oxides of manganese. H. H. B. S.

The Advantage of using "Normal Volumes" in Analysis. M. Monhaupt. (*Chem. Zeit.*, 1898, xxii., 806.)—In the ordinary routine work of a laboratory, it is customary to weigh out such a quantity of the substance under examination that the final figures (weights, volumes, etc.) shall give at once the percentage of the ingredient sought; the author pleads that the same device should be adopted when the sample is measured at the commencement of the test. For instance, in the potash trade, where determination of the proportion of potassium chloride in raw products, etc., is constantly required, the original ("normal") weight is always a multiple of 0·3056 gramme—the factor which converts K_2PtCl_6 into KCl—and the final weight multiplied by 100 is the percentage desired. Similarly, when the analysis has to be conducted on a liquid, 30·56 c.c. (the "normal volume") may advantageously be measured in a special pipette, and tedious calculation be avoided here also.

F. H. L.

APPARATUS.

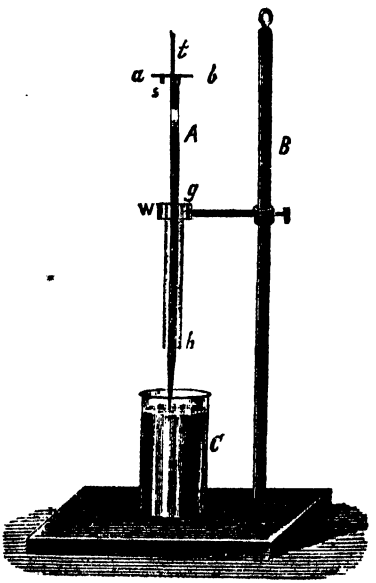
Apparatus for the repeated Extraction of Liquids with Immiscible Solvents. T. Posner. (*Chem. Zeit.*, 1898, xxii., 868.)—The idea of this apparatus is to avoid the loss of material which often accompanies the use of a separating



funnel when a solution has to be extracted more than once with the same liquid. It is essentially a combination of two separating-funnels joined together by means of the stop-cock β. The plug of the latter has two passages lying in the same plane: one serves to place A and B in connection, the other simultaneously allows the air in B to escape—the bulb shown in the enlarged section being intended to catch any splashes. The liquid to be extracted is placed in A; if the solvent is specifically heavier, it is simply run off into B, which acts as a reservoir, while a fresh portion is brought into A and agitated once more. When the solvent is lighter than the liquid, extraction takes place in A, the aqueous portion is passed into B, A is emptied into a beaker or flask, the whole apparatus is inverted, and the liquid run back into A for treatment again. To prevent mistakes one end of the crutch handle of β is made of coloured glass.

F. H. L.

Apparatus for Determining the Consistency of Grease and Similar Articles. **R. Kissling.** (*Chem. Zeit.*, 1898, xxii., 867.)—This is an improved form of the apparatus originally devised for estimating the viscosity of a grease by noting the time occupied by a weighted rod to sink into the mass. A is an aluminium rod weighing exactly 50 grammes, 300 mm. long, 9 mm. in diameter, and tapered from a point 55 mm. above its lower extremity to its end. ab is an X-shaped plate of brass fastened to the rod, having a pin t, on which three annular weights of 50, 100, and 200 grammes respectively can be dropped; s is a stop that at the end of the test rests upon W. gh is a glass tube 150 mm. long and 9·5 mm. in internal diameter, supported vertically by the metal ring W. C is filled with the material under examination to a height of 120 mm. When making a test, it is desirable so to arrange the apparatus that the rod shall fall in between 10 and 60 seconds; if this be done, by adding the number of seconds to the total weight of A (including the brass weights, if one or more be employed), a "consistency number" is given directly. But whenever the period of fall exceeds 100 seconds, if the weight of A is 50 grammes the number of seconds must be divided by ten, and if the weight is 100 grammes divided by two before adding the time to the weight. In this manner greases and gelatinous preparations can be valued simply and with fair accuracy, for the apparatus is capable of differentiating such bodies according to their viscosity into materials having "consistency numbers" ranging between 94 and 405.



F. H. L.

THE ANALYST.

FEBRUARY, 1899.

PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS.

An extraordinary general meeting was held on Wednesday, January 4, 1899, in the Rooms of the Chemical Society, Burlington House, Piccadilly, for the purpose of amending the constitution and rules of the Society.

The President (Dr. Dyer), who occupied the chair, explained the amendments suggested by the council, which, in addition to minor alterations, included a re-modelling of the conditions under which candidates might apply for admission to the membership of the Society. The chief amendment in the rule bearing upon this point was in the omission of the hitherto existing provision that candidates for membership should be analysts in actual practice, a qualification which it had always been difficult to define, and one which it had been frequently necessary to interpret with some degree of elasticity. Notwithstanding this, the rule had formed a technical barrier to the inclusion within the ranks of the Society of a number of qualified analysts who otherwise would have been glad to join the ranks of the Society. The council believed that the new and wider definition would, by removing this difficulty, be advantageous to the Society, as well as to the profession from the ranks of which its members were drawn.

The adoption of this new definition of qualification would render the existence of the class of associates, viz., assistants to analysts in actual practice, no longer necessary, and it was therefore proposed that the Society should not in future include associates. The present associates who had attained the age of twenty-one would at once be eligible for admission to full membership by election in the usual way, while the remainder, whose number, he believed, was but very small, would become eligible on reaching that age.

After a discussion, in which Messrs. A. E. Ekins, E. W. Voelcker, S. Rideal, E. J. Bevan, H. Droop Richmond, C. H. Cribb, and Leo Taylor took part, the new constitution and rules were unanimously adopted, on the motion of the President, seconded by Mr. A. E. Ekins.

The monthly ordinary meeting of the Society was held on Wednesday, January 4, 1899, after the extraordinary meeting, the President (Dr. Dyer) being in the chair.

The minutes of the previous ordinary meeting were read and confirmed.

Mr. F. G. J. Bird, analyst to Messrs. C. R. Harker, Stagg and Morgan, London,

and Mr. J. H. Heywood, Heywood (Lancs.), were proposed for election as members.

On the proposition of Dr. Rideal, seconded by Mr. A. C. Chapman, Mr. B. Kitto and Mr. C. H. Cribb were appointed auditors of the Society's accounts for the past year.

The following papers were read:

"The Occurrence of Barium Compounds in an Artesian Well Water," by John White;

"A New Test for Formaldehyde," by Norman Leonard, B.Sc., and Harry M. Smith;

"On Caper Tea," by Charles Estcourt; and

"The Adulteration of Sweet Spirit of Nitre with Potassium Nitrate," by W. F. Lowe, A.R.S.M.

Messrs. Lester Reed, F.I.C., Harry Broadbent, F.I.C., W. T. Burgess, F.I.C., and T. S. Goodwin were elected members of the Society.

ON CAPER TEA.

By Charles Estcourt, F.I.C.

(*Read at the Meeting, January* 4, 1899.)

In the early days of the Food Adulteration Act it was found that tea of all kinds was largely adulterated. Caper tea especially was found to show evidence of gross fraud on the part of the Chinese growers, and as this kind of tea was much used in the North, and especially in Lancashire, I then examined a large number of samples, the analyses of which demonstrated that this tea, as then sold, contained quartz and magnetic iron ore nodules in quantities varying from 1 per cent. up to as much as 35 per cent. Other analysts also turned their attention to caper tea, the result being that gross adulteration was practically stamped out, as shown by the very few cases of caper tea certified by public analysts as adulterated since 1875. Out of a large number of samples of caper tea analysed in my laboratory from 1876 to 1897, only one contained more than 1 per cent. of mineral impurity. Mr. G. W. Wigner, in 1875, analysed a number of samples of caper tea drawn from bond. The lowest total ash that he found was 5·75 per cent., and the highest 7·02 per cent. The ash insoluble in hydrochloric acid varied from 0·70 per cent. to 1·67 per cent. In seven out of the nine cases which he records the ash insoluble in hydrochloric acid was less than 1 per cent. Dr. Bell, in 1881, in two samples of caper tea from bond, found that the total ash (calculated on the dry tea) was 6·38 and 6·63 per cent., and the ash insoluble in hydrochloric acid 0·68 and 0·93 per cent. In 1882 Mr. Carter Bell published in the Analyst the results of analyses of seventeen samples of caper tea purchased in Salford. The total ash of nine of these was less than 7 per cent., in only three cases exceeded 7·5 per cent., and in only one case exceeded 8 per cent.

Dr. Bell, the late Principal of the Inland Revenue Laboratory, in his book on "The Chemistry of Foods," says, in reviewing his analyses of genuine imported teas,

"the percentage of ash in no case, except one, . . . reaches 8 per cent. . . ." on the dried tea. This last ash, when calculated on the original undried sample, is equivalent to 7·7 per cent. With regard to mineral adulterants, Dr. Bell says, speaking of tea generally: "If the insoluble ash exceeds much more than 1 per cent., there is evidence of the addition of either sand, quartz, or other earthy impurities."

On August 24, 1898, I certified a caper tea as containing "2·25 per cent. of sand and ferruginous earth." At the hearing of the summons, the magistrates, without my analysis being questioned, sent the third portion of the sample to Somerset House. The following certificate (which, it will be observed, is not signed by either the Principal or Deputy Principal), was in due course received:

"GOVERNMENT LABORATORY,
"Clement's Inn Passage,
"Strand, London, W.C.

"The sample of caper tea marked 532, and referred to in your letter of the 7th instant, was received here on the following day securely sealed. We hereby certify that we have analysed the tea with the following results:

Total mineral matter	7·50	per cent.
Mineral matter soluble in water	2·36	,,
,, ,, in acid	3·12	,,
Silica (less sand)	1·38	,,
Sand	0·64	,,

"With the view of ascertaining how far the sample in question differs from caper tea now met with in commerce, and of fair commercial quality, we have procured a number of authentic samples of such teas out of bond. On analysis they furnish the following results:

Per Cent.	A.	B.	C.	D.	E.	F.	G.
Total mineral matter ...	6·74	7·90	7·26	7·20	6·44	7·08	8·00
Soluble in water ...	2·58	2·38	2·30	2·54	2·14	2·46	2·30
Soluble in acid	2·98	2·84	2·90	2·88	3·08	2·14	4·06
Silica (less sand) ...	1·04	0·32	1·76	1·40	1·06	1·26	1·08
Sand	0·14	2·36	0·30	0·38	0·16	1·22	0·56

"It is evident that the amount of foreign mineral matter in the case of the sample referred to us is not higher than that which may be present in genuine caper teas. We are of opinion, therefore, that the small proportion of extraneous matter present has become unavoidably mixed in the process of collection and preparation.

"As witness our hands this twenty-first day of September, 1898.

"(Signed) "G. LEWIN.
"H. W. DAVIES.

"THE CLERK TO THE MAGISTRATES,
"City Police Court, Manchester."

The summons was dismissed.

It will be seen that in the certificate the whole question of what genuine caper tea is, is begged. Samples of tea are taken out of bond and are assumed to be *authentic*, or (as in the term used lower down in the Somerset House certificate)

These teas, as compared with those imported in 1875, appear to me to be genuine only inasmuch as they have not been stopped by the Customs, but have been, improperly, allowed to go into consumption. The proportion of mineral matter insoluble in hydrochloric acid—not stated as a separate item in the official analyses, but calculated by deducting the water-soluble and acid-soluble ash from the total—would be as follows:

	A.	B.	C.	D.	E.	F.	G.
Mineral matter insoluble in hydrochloric acid ...	1·18	2·68	2·06	1·78	1·22	2·48	1·64

Deduced in the same way, the ash insoluble in hydrochloric acid found at the Government Laboratory in the third portion of my sample was 2·02 per cent. It will be seen that in these teas, taken from bond, in three cases the ash insoluble in hydrochloric acid exceeded 2 per cent., being in one case 2·68 per cent., and in another 2·48 per cent.; whereas Dr. Bell, the former Principal of the Inland Revenue Laboratory, laid down the dictum that if the insoluble ash much exceeded 1 per cent., there was evidence of the addition of either sand, quartz, or other earthy impurities. In my sample I found nodules of quartz rolled up in the leaves, and therefore cannot consider that the quartz had been, as is suggested in the certificate from the overnment Laboratory, unavoidably mixed with the tea. It appears desirable that public analysts should be aware of the fact that the limit of earthy impurity fixed by Dr. Bell is no longer adopted at the Inland Revenue Laboratory, and that, apparently, any sample of caper tea containing not more than 2·68 per cent. of ash insoluble in hydrochloric acid is to be regarded as genuine.

Attention may also be directed to the proportion of ash soluble in water in caper tea. Mr. Wigner, in 1875, found the proportion of ash soluble in water to vary from 2·64 to 3·66 per cent. in caper tea, the average of nine samples showing 3·07 per cent. In Dr. Bell's two samples, analysed in 1881, the proportion of ash soluble in water, on the dry tea, were 3·21 and 3·47 per cent., equivalent to 2·98 and 3·23 per cent. in the moist tea. Since the sample which gave rise to this paper contained, according to the analysis made in the Government Laboratory, only 2·36 per cent. of mineral matter soluble in water, it might, on the basis of the foregoing figures, be reasonably regarded as containing exhausted leaves. But the figures found for soluble ash in the seven samples quoted in the report from the Government Laboratory as authentic samples of caper tea out of bond range from 2·58 down to 2·14 per cent.; and if these figures are to be accepted as fixing a standard of genuineness in the matter of the admixture of exhausted leaves, the limit of 3 per cent. virtually adopted by Dr. Bell presumably also disappears.

RAPID ESTIMATION OF SMALL QUANTITIES OF FREE PHOSPHORUS IN PHOSPHORUS PASTES, ETC.

BY LESTER REED, F.I.C.

WHEN a solution of bromine in carbon disulphide is added gradually to one of phosphorus in the same liquid, the colour of the bromine is discharged almost instantly until a certain point is reached. At this point the phosphorus and bromine consumed appear to stand to each other in the proportion $P : Br_3$. If, however, a small quantity of alcohol containing a little water is present, the reaction is equally definite, but the proportion appears then to be $P : Br_5$, the end of the reaction in this case being when the alcoholic layer retains a decided yellow colour for a minute or two. The reaction does not appear to be at all affected by the presence of wheat-flour or glucose.

To apply this principle to the estimation of free phosphorus in phosphorus paste, we can proceed as follows: A solution of bromine is to be prepared containing from 5 to 10 grammes in 50 c.c. of carbon disulphide. This is standardized by a weighed quantity of yellow phosphorus dissolved in about 20 c.c. of carbon disulphide. To do this a stoppered bottle, such as is used for the determination of the hardness of water, is taken, and in it is placed about 1 c.c. of paste, made of flour, glucose, and water, then 5 c.c. of absolute alcohol (which should be shaken with the paste till it is disintegrated), and then the phosphorus solution, thus imitating the conditions of the actual estimation to follow. The bromine solution is now added from a burette in small quantities at a time, with shaking, until a decided and permanent yellow colour is obtained in the floating layer.

A weighed quantity of the sample of phosphorus paste is then taken in the same or a similar bottle, 5 c.c. of absolute alcohol added and well shaken until the paste is thoroughly disintegrated, 20 c.c. of carbon disulphide are then added, and the titration proceeded with as above. The method of calculating the result is, of course, obvious.

In the presence of phosphites, resulting from the slow oxidation of phosphorus, an allowance would have to be made for the quantity of bromine solution decolorized by a filtered extract of the paste made with cold 50 per cent. alcohol.

I desire to submit this process quite tentatively, although the results appear, from my own experiments, to be reliable when the conditions are carefully observed. Its extreme simplicity and rapidity seem to me strong points in its favour. I should be very pleased to hear of anyone's experience with it.

ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

FOODS AND DRUGS ANALYSIS.

Determination of Dirt in Milk. R. Eichloff. (*Zeit. für Untersuch. der Nahr. und Genussmittel*, 1898, 678-683.)—The following method is proposed: 300 grammes of milk are weighed and transferred to eight test-glasses 15 cm. long by 1·5 cm. wide. These are then placed in a centrifugal apparatus, which is rotated at a speed of about

thirty revolutions a minute for five minutes. The dirt settles completely to the bottom. The cream and the greater part of the milk are now carefully removed, leaving the sediment undisturbed, and the remainder of the milk with the sediment is then washed out into a fresh tube and again centrifuged. The sediment is finally filtered off, with the help of a filter-pump, through a dried and weighed asbestos filter-tube, which is then thoroughly washed, dried, and re-weighed. H. H. B. S.

On the Changes which take place in Milk Fat during the Ripening of Cheese. A. Kirsten. (*Zeit. für Untersuch. der Nahr. und Genusmittel*, 1898, 742.)—The results obtained are shown in the following table. They are in every case the mean of duplicate experiments:

	Date.	Number Days Ripening.	Refractometer Number at 25° C.	Reichert-Meissl-Wollny Number (c.c. $\frac{N}{10}$ BaO for 5 Grammes of fat).	Köttstorfer Number (Milligrammes KHO required for saponifying 1 Gramme).	Hehner Number (Insoluble Fatty Acids from 100 Parts of Fat).	Remarks.
			I.—Fat from Camembert Cheese.				
1	17, 5, '96	0	53·0	27·1	227	87·6	
2.	27, 5, '96	10	53·1	27·1	227	87·2	
3.	3, 6, '96	17	52·7	26·6	226	87·7	
4.	10, 6, '96	24	52·7	26·6	225	87·4	Cheese ripe.
5.	17, 6, '96	31	52·4	26·9	225	87·4	Cheese over-ripe.
			II.—Fat from Neufchâtel Cheese.				
1.	15, 9, '96	0	53·7	29·1	226	87·3	
2.	2, 10, '96	18	53·7	28·4	225	86·8	
3.	15, 10, '96	31	53·6	26·0	223	86·5	Cheese ripe.
			III.—Fat from Tilsit Fat Cheese.				
1.	25, 2, '97	0	52·2	27·9	226	87·7	
2.	24, 8, '97	181	52·1	27·3	224	87·4	Cheese ripe.
			IV.—Fat from Dutch Cheese.				
1.	7, 1, '97	0	53·1	27·5	229	88·4	
2.	3, 11, '97	300	52·4	27·5	227	88·1	Cheese ripe.

H. H. B. S.

Detection of Cane-Sugar in Wine, Saccharine Liquids, etc. G. Papasogli. (*Giorn. di Farm. di Trieste*, 1898, iii., 301; through *Chem. Zeit. Rep.*, 1898, 320.)—An aqueous solution of cane-sugar gives a fine permanent amethyst colour when it is mixed with a cobalt salt and a small excess of caustic soda, and the tint is visible even in 0·05 per cent. solutions. Grape-sugar yields a blue which rapidly bleaches

and finally becomes dirty green. Dilute glycerin only produces a faint green. Solutions containing 20 per cent. of alcohol, 10 per cent. of cane and 10 per cent. of grape sugar respectively, coloured with caramel, were prepared. 15 c.c. were decolorized with 0·5 gramme of animal charcoal, filtered, treated with 0·5 c.c. of a 5 per cent. solution of cobalt nitrate, shaken, and then 2 c.c. of 50 per cent. caustic soda were added: the reactions appeared in every case. The amethyst colour characteristic of cane-sugar can be recognised even when the sugars are composed of 9 parts of grape to 1 of cane sugar. Wines must be bleached with lead acetate to remove colouring matter and tartaric acid. Condensed milk may be diluted with twice its volume of water, and treated with lead acetate. Gum and dextrin must be removed either with the normal or ammoniacal acetate of lead. F. H. L.

The Detection of Starch in Black and White Mustard Seed. J. W. Lloyd. (*Amer. Jour. Pharm.*, 1898, lxx., 433-439.)—The ordinary method of testing for starch by adding iodine solution to the aqueous decoction may fail with black mustard, owing to the liberation of mustard oil when the mustard comes in contact with water. This oil combines with the iodine, and when only a small quantity of starch is present no blue colour is obtained. The author has found that copper sulphate has the property of preventing the formation of the oil of mustard even when used in a solution of 0·2 per cent. strength, and also that on boiling the mustard seed with a solution of potassium iodide the same result is attained.

C. A. M.

The Estimation of Gelatin in Gums and Food Substances. A. Trillat. (*Ann. de Chim. Anal. Appl.*, 1898, iii., 401, 402.)—The property which formaldehyde possesses of forming insoluble compounds with proteid substances was applied by Beckmann (ANALYST, xx., 44) to the estimation of gelatin and albumin in peptones. A similar process is here advocated for the detection and estimation of gelatin in general, and especially when mixed with gums. The substance under examination is dissolved in water, the insoluble matter removed by filtration or decantation, and the solution evaporated to the consistence of a syrup. About 1 c.c. of commercial formalin is then added, and the evaporation continued till the mass becomes pasty. The residue is taken up with boiling water, which dissolves the gum and leaves the insoluble gelatin compound. After standing for twenty-four hours, the clear or slightly opalescent supernatant liquid is decanted, and the precipitate washed with boiling water, dried on the water-bath, and weighed.

In a test analysis of a mixture consisting of 85 grammes of gelatin, 20 grammes of gum arabic, and 10 grammes of sugar, the gelatin was determined within 1 per cent. of the actual quantity.

The gelatin of the jellies of commerce is determined in a similar manner. In all cases the substance should be brought to a pasty consistence before adding the formaldehyde, since in a dilute solution no precipitate is obtained. C. A. M.

Glazed Coffee. E. Hanausek. (*Oesterr. Chem. Zeit.*, 1898, i., 482.)—A new substance for glazing coffee-berries, in order (so the makers assert) to prevent loss of aroma and of caffeine, and improve the material commercially, chemically, and physiologically, consists of shellac. This is being put on the German and Austrian markets in small angular orange fragments by G. Schneider Nachfolger, of Ludwigshafen a/R., and is to be employed in amounts of 0·5 to 1·0 per cent. according to the time of preservation desired. F. H. L.

The Occurrence of a Ptomaine in Coffee. S. Bein. (*Zeits. angew. Chem.*, 1898, 658-661.)—A sample of coffee which had caused symptoms of poisoning in the members of a family which had drunk the infusion was examined by the author. It was found to contain none of the usual metallic or alkaloidal poisons, and was free from artificial beans. It consisted of a mixture of black, over-burnt beans, including fragments of husks, etc., with about 18 per cent. of beans of a lighter colour, containing about 18 milligrammes of caffeine in 100 grammes. The black part of the mixture contained no caffeine, and had a coating on the surface in which was 0·42 per cent. of sodium chloride.

On testing, the coffee gave indications of the presence of a ptomaine.

As to the origin of the ptomaine, the following explanation is suggested: There are in commerce a number of worthless products, consisting of mixtures of various kinds of spoilt coffee, such as that damaged by sea-water, etc., and known as "*Triage.*" These are commonly mixed with a small proportion of a better substance, in order to render the article saleable as coffee. Since normal coffee only contains about 0·01 per cent. of sodium chloride, the author regarded the presence of the large percentage of that salt in the black part of the mixture as proof of its having been damaged by salt-water in transit. If such coffee were then stored in a damp condition in a badly-ventilated room, a decomposition of the proteid substance of the bean would soon be brought about.

That under these circumstances ptomaines may be produced was proved experimentally.

The authors suggest as a further cause, the roasting of the damaged coffee until black, with the object of concealing its decomposed appearance. On over-roasting, deep-seated changes occur; there is decomposition of the fat, of the proteids, of the coffee-tannin, and of the woody fibre, and bodies with objectionable smell are produced in considerable quantity. From the proteids especially pyrrol substances are derived, and these do not escape completely, but are partially condensed on the beans. The caffeine also is destroyed, and methylamine and its derivatives are formed in sufficient quantity to impart their characteristic objectionable odour.

From an experimentally over-roasted coffee the author prepared an aqueous extract, from which he succeeded in isolating pyrrol bodies and amine bases. The fact that no caffeine was found in the black portion of the coffee mixture can thus be accounted for by too high a roasting. The author points out that these products of over-roasting (amine bases pyrrol derivatives, etc.) are capable of producing very similar symptoms in animals to those produced by the products of putrefaction, even when in very dilute condition. C. A. M.

An Adulterated Gamboge. J. F. Woolsey. (*Amer. Jour. Pharm.*, 1898, lxx., 446, 447.)—Powdered gamboge as usually obtained is of a bright orange yellow colour, and contains 70 to 80 per cent. of resin, 3 to 4 per cent. of ash, 4 to 6 per cent. of moisture and gum. A good gamboge contains 75 per cent. of resin. The trace of starch usually met with is not regarded as an adulteration, and is due to the method of collecting and packing.

The author has recently met with a sample of a dull ochre colour, which was heavily adulterated. On treatment with 95 per cent. alcohol less than 40 per cent. dissolved. The added substance was found to consist of starch or flour (*cf.* ANALYST, xxi., 266). C. A. M.

ORGANIC ANALYSIS.

Detection of Iodoform in Aqueous Fluids. L. von Stubenrauch. (*Zeit. für Untersuch. der Nahr. und Genussmittel*, 1898, 737.)—The method proposed by the author rests upon the following experiment. Two equal portions (one or two crystals) of iodoform are mixed into an emulsion with equal quantities (3 to 5 c.c.) of water. One portion is reduced by warming with zinc dust and a drop of acetic acid and afterwards filtered, whilst the other portion is left unreduced. On now adding a single drop of nitric acid and a little starch solution to each portion, a blue colour is produced in the reduced portion, but not in the other. It is necessary to limit the nitric acid added to a single drop. If a large quantity be used, the iodoform is decomposed and the blue colour produced without the reduction with zinc dust and acetic acid. On applying the test, if a blue colour be produced on addition of a drop of strong nitric acid and some starch, potassium iodide, hydriodic acid, or a soluble, easily decomposable organic compound of iodine may be present; but if, on the contrary, the blue colour appear only after reduction with zinc dust and acetic acid, the presence of iodoform is indicated. A solution of iodoform in water behaves in the same way as an emulsion. A difficulty, however, arises when hydriodic acid, an iodide of an alkali metal, or a soluble organic compound of iodine, is present in conjunction with iodoform. In this case the test is only available when the proportion of the other bodies to the iodoform is relatively small, so that the difference in the depth of the blue colour produced before and after reduction is distinctly apparent. The presence of albumin, except in very small quantities, also interferes with the test.

The author's experiments with the method show that iodoform taken internally is not excreted as such in the urine, but is converted into an organic iodine compound or into an iodide of an alkali metal. H. H. B. S.

Preparation of a Standard Solution of Ethyl Aldehyde. X. Rocques. (*Annal. de Chim. Anal.*, 1898, iii., 365.)—The preparation of an alcoholic solution of aldehyde for use as a standard in the colorimetrical determination of aldehyde is not an easy matter, owing to the readiness with which polymerization occurs and the difficulty of obtaining a pure substance.

The author obviates this difficulty by using aldehyde ammonia $CH_3 - CH\begin{matrix}HO\\NH_3\end{matrix}$

as the starting-point. This substance, as met with in commerce, is in the form of rhombohedral crystals melting at 70° to 80°. It is very soluble in alcohol, is insoluble in ether, and is decomposed by dilute acids with the re-formation of aldehyde. It is purified by trituration in a mortar with successive portions of anhydrous ether, the liquid being decanted each time. The final residue is dried in the air, and afterwards *in vacuo* over sulphuric acid.

1·386 grammes of the dry compound are dissolved in about 50 c.c. of pure alcohol (96 per cent.), and 22·7 c.c. of normal sulphuric acid (mixed with more of the same alcohol) added, an immediate precipitate of ammonium sulphate being formed. The liquid is made up to 100 c.c. with the alcohol, and an additional 0·8 c.c. added to compensate for the volume of the ammonium sulphate. It is then shaken, allowed to stand overnight, and filtered. The filtrate contains 1 per cent. of aldehyde, and is diluted with the necessary quantities of water and 50 per cent. alcohol, so as to obtain a solution containing 50 milligrammes in a litre of 50 per cent. alcohol. C. A. M.

Reaction between Ferric Chloride and Phenol in presence of Alcohol. F. Peters. (*Zeits. angew. Chem.*, 1898, 1078.)—It is generally believed that the violet colour produced when ferric chloride is mixed with an aqueous solution of phenol does not appear if the latter be dissolved in alcohol. The author finds that on dropping a 10 per cent. aqueous solution of ferric chloride into a solution of phenol in dilute spirit, the colour is not prevented from appearing if the proportion of alcohol does not exceed 3·19 v/v, or 2·53%; but as the amount rises to 3·44 v/v, or 2·73%, the reaction becomes indistinct, and above that limit it does not occur at all.

F. H. L.

Reactions of Some Common Phenols. G. Deniges. (*Bull. Soc. Pharm. Bordeaux*, 1898, 241; through *Ann. de Chim. Anal.*, 1898, iii., 381, 382.)

Reactions with Mercuric Sulphate.—Five centigrammes of the polyphenol are dissolved in 2 c.c. of water, 3 c.c. of the author's mercuric reagent (ANALYST, xxiii., 216) added, and the whole shaken.

A lemon-yellow precipitate is obtained	Pyrogallol.
A yellowish-white ,, ,, ...	Phloroglucinol.
No precipitate. The liquid becomes yellow, changing to yellowish-red and to reddish-brown	Pyrocatechin.
No precipitate. The liquid remains colourless, or has a slight greenish-yellow tint	Hydroquinone.
On boiling becomes yellowish-red	
On boiling no perceptible change	Resorcin.
After immersion of the tube in cold water and prolonged standing a yellowish precipitate	

Reactions with Soda.—From 15 to 20 centigrammes of the substance are dissolved in 3 to 4 c.c. of alcohol, 1 c.c. of a solution of soda allowed to flow down the side of the tube and the zone of contact observed.

(*a*) A coloured zone is produced immediately.		
Red	surmounted by a green ring. On shaking the liquid becomes green	Hydroquinone.
Red	surmounted by a white ring. On shaking the liquid becomes brown	Pyrogallol.
Yellow, with a yellow coloration of the supernatant alcohol		Pyrocatechol.
White		Phloroglucinol.
(*b*) No immediate coloration, but after some time a green ring appears		Resorcin.

(*Cf.* ANALYST, xxi., 295.) C. A. M.

Humble-Bees' Wax. E. E. Sundvik. (*Zeits. Physiol. Chem.*, 1898, xxvi., 56; through *Chem. Zeit. Rep.*, 1898, 321.)—From 130 grammes of comb the author obtained, by extraction with ether or chloroform, about 30 grammes of crude wax, melting between 35° and 40° C. The odour was pleasant, resembling honey, and not at all rancid. The wax bleached almost perfectly when exposed to daylight in thin layers; but became very rancid in smell. Treated with weak caustic potash, and crystallized six or seven times from alcohol, it melted at 69°-70°; finally it melted at 74° or 75° C. The intermediate product possessed the sticky nature of ordinary beeswax; the purest did not. The crystals form fine, soft, woolly needles. Even in the cold it is more or less soluble in the regular fat-solvents. Its empirical formula is $C_{34}H_{70}O$. Heated to 150° or 160° with benzoic anhydride, it gives a substance easily soluble in hot or cold alcohol, which, after several recrystallizations, melts at 55° C.

F. H. L.

The Estimation of Pentoses. A. Grégoire and E. Carpiaux. (*Bull. de l'Ass. Belge*, 1898, xii., 143-151.)—This paper gives an account of the authors' investigation on the different methods of determining pentoses, and describes various modifications which they have adopted.

For the estimation of pentoses, after conversion into furfural, two methods are employed: that of Tollens, in which the furfural is precipitated by means of phenylhydrazine; and that of Councler, in which phloroglucol is used as the precipitant. Several chemists have been unable to obtain satisfactory results by the second method, and hence the precipitation of the pentoses as hydrazones is the one in general use:

$$C_5H_4O_2 + C_6H_5NH.NH_2 = C_6H_5NH.NH.C_5H_3O + H_2O.$$

The hydrazone was formerly determined volumetrically (Tollens and Günther) or gravimetrically (Tollens and Chalmot), but Flint and Tollens' recent researches (*Landw. Vers. Stat.*, xlii., 395) have shown that in the volumetric method the distillation of the substance with hydrochloric acid yields small quantities of substances (acetone, etc.), which combine with the phenylhydrazine, although they do not form an insoluble compound, and hence the results are too high.

In the gravimetric method the distillate containing the furfural is diluted to 400 c.c. with hydrochloric acid, neutralized with sodium carbonate, slightly acidified with acetic acid, the furfural precipitated with an acetic acid solution of phenylhydrazine, the solution brought to 500 c.c., and, after being mechanically stirred for thirty minutes, the precipitate collected on a filter of glass wool, washed with water,

dried in a current of air, and weighed. The weight of the hydrazone multiplied by 0·538 gives the furfural.

The authors find that there is a slight source of error in this method, owing to the hydrazone dissolving to a slight extent in the water required to wash the precipitate. To obviate this they have devised a gas-volumetric process, in which the nitrogen in the phenylhydrazine is determined before and after the precipitation, and the difference calculated into the amount taken up by the furfural.

The original phenylhydrazine is decomposed by means of copper sulphate :

$$C_6H_5NH.NH_2 + 2CuSO_4 = H_2SO_4 + C_6H_6 + N_2 + Cu_2SO_4,$$

but this is not possible in the presence of sodium chloride, as in the filtrate from the precipitated hydrazone, unless the process is modified. Phenylhydrazine acetate, like ammonium acetate, decomposes on boiling, but the authors' experiments show that by making the liquid strongly acid with hydrochloric acid, the phenylhydrazine can be boiled without alteration.

In the gas-volumetric process finally adopted the nitrogen in the phenylhydrazine reagent was determined in the following manner : From 4 to 4·5 grammes were dissolved in 250 c c. of water, 25 c.c. of the solution were mixed with 20 c.c. of concentrated hydrochloric acid, boiled for several minutes to remove all air, and introduced while hot into a Schloesing's (nitric acid) apparatus containing 25 c.c. of a 20 per cent. solution of copper sulphate and several c.c. of hydrochloric acid, from both of which all air had been expelled by boiling. The reaction took place instantaneously, in accordance with the equation :

$$C_6H_5N.NH_2 + 4CuSO_4 + HCl = 2H_2SO_4 + C_6H_5Cl + 2Cu_2SO_4 + N_2.$$

The liberated nitrogen was collected and measured in the usual manner, corrections made for the vapour tension of water and chlorbenzene at the temperature of observation, and the calculated weight of nitrogen multiplied by 3·857. Thus in a typical case 94·8 c.c. of gas were obtained at a temperature of 19° C. and a pressure of 748 m.m., which corrected for temperature, pressure, tension of the water vapour, and chlorbenzene vapour was 721 m.m., corresponding with 88·51 per cent. of the phenylhydrazine taken.

For the precipitation of the furfural, 81·5 grammes of sodium chloride (the quantity corresponding to the amount of hydrochloric acid which distils over in the estimation of pentoses) were dissolved in about 400 c.c. of water, 2 drops of acetic acid added, and a quantity of furfural not exceeding 0·6 gramme. After the addition of an *aqueous* solution of phenylhydrazine (4 or 4·5 grammes in 250 c.c.), the liquid was made up to 500 c.c., and shaken for at least an hour in a Witt agitator.

The filtrate was at once mixed with hydrochloric acid, which was found to prevent the decomposition of phenylhydrazine acetate. It was then boiled to expel the air, and 200 c.c. containing 20 c.c. of hydrochloric acid were introduced into Schloesing's apparatus, and the nitrogen determined as before and deducted from the amount found in the first determination. The result multiplied by the factor 3·429 gave the amount of furfural.

In seven analyses of furfural by this method the percentages found varied from 99·4 to 102·4 per cent., the mean being 100·5 per cent. C. A. M.

A Method of Analysing Natural and Artificial Organic Colouring Matters. **A. R. Rota.** (*Chem. Zeit.*, 1898, 437-442.)

I. *Identification of Individual Colouring Matters.* — In this paper the author draws up a scheme of analysis based upon the recent views of Nietzki, Witt, Armstrong, and others, as to the relationship which exists between the constitution and colour of these substances.

Regarding organic colouring matters as quinone derivatives (Armstrong, Nietski), those which are based upon mono- and di- amido-quinone are reducible by stannous chloride; while those which are regarded as derivatives of a quinone, in which an oxygen atom is replaced by a di-valent hydrocarbon group in the quinone ring, are not reducible. Thus, if O = R = O represent an ortho- or para- quinone, the nitroso- azo- and imido- quinone colour derivatives are reducible:

$$\underset{\text{oximido-quinone}}{O=R=N-} \quad \text{or} \quad \underset{\text{di-imido-quinone,}}{-N=R=N-}$$

but not such derivatives as oxyquinone- and triphenyl- methane colouring matters:

$$\underset{\text{oxycarbo-quinone}}{O=R=C=} \quad \text{and} \quad \underset{\text{imido-carbo-quinone.}}{-N=R=C=}$$

The reduced colouring matters can be subdivided into two groups, according as to whether the colour is restored on oxidation with ferric chloride or atmospheric oxygen; and the unreduced colouring matters can also be subdivided according to their behaviour on treatment with caustic potash. Four main groups are thus obtained, as shown in the subjoined scheme.

The aqueous or alcoholic solution of the substance is diluted to about 1 : 10,000, and 5 c.c. treated with 4 to 5 drops of concentrated hydrochloric acid and about the same quantity of a 10 per cent. solution of stannous chloride. The mixture is shaken, and, if necessary, warmed to the boiling point. If no decolorization occurs, the solution of the colouring matter should be again tested with stannous chloride after still further dilution.

A.

Classification of Organic Colouring Matters.

A portion of the aqueous or diluted alcoholic solution is treated with HCl and $SnCl_2$.

Complete decolorization. Reducible colouring matters. The colourless solution is treated with Fe_2Cl_6, or shaken with exposure to the air.		The colour is changed no further than with HCl alone. Non-reducible colouring matters. A part of the original solution is mixed with 20 per cent. KOH and warmed.	
The liquid remains unchanged. Colouring matters not reoxidizable.	The original colour restored. Reoxidizable colouring matters.	Decolorization or a precipitate. Imido-carbo-quinone colouring matters.	No precipitation. The liquid becomes more coloured. Oxy-carbo-quinone colouring matters.
Class I.	Class II.	Class III.	Class IV.
Nitro-, nitroso-, and azo-colouring matters, including azoxy- and hydrazo- colours.	Indogenide- and imido-quinone colouring matters.	Amido-derivatives of di- and tri- phenylmethane, auramines, acridines, quinolines, and colour derivatives of thiobenzenil.	Non-amide diphenylmethane colouring matters, oxy-ketone colouring matters (most of the natural organic colouring matters).

The individual colouring matters in the four main groups may often be identified by reference to one of the published tables of their physical, chemical, and tinctorial properties, which, however, only describe a part of the dyes now met with in commerce. In doubtful cases resort must be made to the spectroscope. A differentiation can often be made in the case of halogen derivatives of similar phthaleins, for instance, by determining the halogen after igniting the colouring matter with lime. The detection of sulphur by fusing the dye with potassium nitrate, and testing the melt for sulphuric acid, often enables one to differentiate between two substances, as, for instance, between the thiazines and oxazines. When the colouring matter was reduced by stannous chloride, the decomposition product may be further examined after removing the tin with sulphuretted hydrogen. Picric acid, for example, gives the colourless tri-amido-phenol, which on treatment with ferric chloride gives the blue amido-di-imido-phenol. The azo colouring matters give on reduction with stannous chloride at least two primary amines in accordance with the equation :

$$R - N = N - R_1 + 2H_2 = R - NH_2 + R' - NH_2 .$$

These amines can often be separated by means of ether. The reduced solution, from which the tin has been removed by means of sulphuretted hydrogen, is treated with caustic potash and the liquid shaken with ether, which dissolves the non-sulphonated amine, and leaves the sulphonated amine in the aqueous layer. The latter can be identified by the characteristic azo-compound which it forms with certain diazo-derivatives. Sulphanilic acid, for instance, obtained by the reduction of naphthol orange, combines with diazo-benzidine-chloride to form a yellow tetrazo-colouring matter. Naphthionic acid yields Congo-red ; and certain oxy-sulphonated amines, such as $C_{10}H_5\begin{cases}NH_2(2)\\OH(8)\\SO_3H(6),\end{cases}$ give a violet colour (diamine black R (C), etc.). Since the NH_2 group resulting from the azo groups must be in the para-position to another amido group in the radicle, a para-diamine is produced, and this can be easily recognised by the thiazin reaction (treatment of the solution freed from tin with hydrochloric acid and ferric chloride in the presence of sulphuretted hydrogen). A para-diamine is also obtained from those colouring matters which contain not the amido group, but two diazo groups, since the middle radicle contains the two azo groups in the para-position. Thus, Sudan III. (A), on reduction with stannous chloride, gives :

$$C_6H_4\begin{cases}N=N-C_6H_5\\N=N-C_{10}H_6(OH)\beta(4)\end{cases}^{(1)} + 4H_2 = C_6H_4\begin{cases}NH_2(1)\\NH_2(4)\end{cases} + C_6H_5.NH_2 + C_{10}H_6\begin{cases}OH(\beta)\\NH_2\end{cases}.$$

By this reaction it is possible in the absence of an amido substance to determine whether a mono-azo or a diazo colouring matter is present.

A scheme of the characteristics of members of the four groups is shown in the following tables :

B.

Class I.—Reduced by $HCl + SnCl_2$ and not Reoxidizable.

Nitro-colouring Matters: $R-NO_2$. Yellow or orange. Soluble in water. Wool and silk dyed directly, but not cotton. The aqueous solution shows tendency to decolorization with HCl. With $HCl + SnCl_2$ partially reduced, giving red nitro-amido derivatives (nitramines) or nitro-phenols turning red in KOH.

- **Nitramines.** Soluble in ether in presence of KOH. $-N=R=N\langle^{O}_{OH}$ *e.g.*, Aurantia.
- **Nitro-phenols.** Insoluble in ether in presence of KOH.
  - *Non-sulphonated.* Soluble in ether in presence of acetic acid. $O=R=N\langle^{O}_{H}$ Victoria yellow.
  - *Sulphonated.* Insoluble in ether. Naphthol yellow.

Nitro-colouring Matters: $O=R=N-OH$. Brown or green, usually insoluble in water. Indirect for fibres. With $H_2SO_4 + C\ HOH$ give blue colour (Liebermann's reaction).

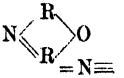
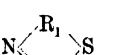
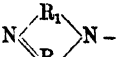
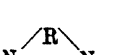
- *Non-sulphonated.* Insoluble in water. Soluble in alcohol. Soluble in ether in presence of acetic acid. Dioxine (L).
- *Sulphonated.* Soluble in water. Insoluble in ether. Naphthol green.

Azo-colouring Matters: $R-N=N-R$. Their aqueous solution decomposed with KOH and extracted with ether, gives an ethereal extract with the annexed characteristics.

- Coloured; shaken with dilute acetic acid yields to it the original colour. **Basic Colouring Matters.**
  - **Non-sulphonated. Amido-azo Colouring Matters.** $-N=R=N-NHR_1$ Bismarck brown.
- Coloured solution; not yielding its colour to dilute acetic acid. **Neutral Colouring Matters.**
  - **Oxyazo-colouring Matters, without Carboxyl.** $O=R=N-HR_1$ Sudan I. (A).
- Colourless solution. Yields nothing to acetic acid. **Acid Colouring Matters.**
  - *Non-sulphonated.* Extracted by ether from dilute solution in acetic acid.
    - **Oxyazo-colouring Matters with Carboxyl Group.**
      - Indirect for cotton wool. Diamond yellow (By).
      - Direct for cotton wool. Chrysamine.
  - *Sulphonated.* Not extracted by ether from solution in dilute acetic acid.
    - **Non-amido Compounds.** Unaltered by HNO_2.
      - Indirect for cotton wool. Bordeaux B (A).
      - Direct for cotton wool. Azo-blue (A).
    - **Amido Compounds.** Changed by HNO_2.
      - Indirect for cotton wool. Solid yellow N (P).
      - Direct for cotton wool. Congo red (A).

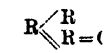
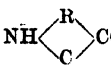
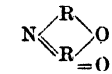
Class II.—Reduced by $HCl + SnCl_2$ and Reoxidizable.

The aqueous or alcoholic solution is treated with KOH and extracted with ether. The ethereal solution washed with water has the annexed characteristics.

- The ethereal solution is coloured or colourless, and yields the original colour to 5 per cent. acetic acid.
  - The solution is readily reduced by $HCl + SnCl_2$ in the cold.
    - **Oxyazines** (no sulphur).  Nile blue A (B).
    - **Thiazines** (sulphur).  Methylene blue.
- **Basic Colouring Matters.** Fixed on wool in alkaline bath.
  - The coloured solution is reduced but slowly and incompletely, even on warming, and with the addition of much $SnCl_2 + HCl$.
    - **Indulines.** Blue colour with conc. H_2SO_4. Blue on dilution.  Induline. Soluble in alcohol.
    - **Safranines.** Green colour with H_2SO_4. On dilution blue, then violet.  Safranine T. Extra (A).

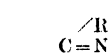
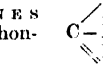
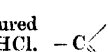
Class II. (continued).—Reduced by $HCl + SnCl_2$ and Reoxidizable.

The aqueous or alcoholic solution is treated with KOH and extracted with ether. The ethereal solution washed with water has the annexed characteristics.

Coloured. Does not yield the colour to acetic acid.	Blue colouring matters changed by HCl on warming.	INDOPHENOLS.		Indophenol.
NEUTRAL COLOURING MATTERS. Insoluble in water. Soluble in alcohol. Fixed on fibres in bath.	Red or blue colouring matters. Unaltered by HCl. With HNO_3 yield isatin.	INDOGENIDES.		Indigotin.
Uncoloured. Yield nothing to acetic acid.	*Non-sulphonated.* Soluble in ether in presence of acetic acid.	OXAZONES.		Fluorescent bl orcein.
ACID COLOURING MATTERS. Soluble in water. Fixed on wool in acid bath.	*Sulphonated.* Insoluble in ether under all circumstances. — Reduced by $SnCl_2$+HCl.	SULPHONATED INDOGENIDES.		Indigo carmine.
		SULPHONATED THIAZINES.		Thiocarmine (C).
	Not reduced by $SnCl_2$+HCl.	SULPHONATED INDULINES.		Soluble nigrosin.

Class III.—Colouring Matters not Reduced by $SnCl_2 + HCl$. Containing the Imido-quinone Carbon Chromophore $-N=R=C=$.

The aqueous or alcoholic solution treated with KOH and extracted with ether.

The ethereal solution is colourless or coloured. The colour is yielded to 5 per cent. acetic acid. BASIC COLOURING MATTERS. Fixed on wool in alkaline bath (NH_3).	Colourless, non-fluorescent, ethereal solution. Yellow colour yielded to acetic acid non-fluorescent. The aqueous solution is decolorized by KOH and decomposed by HCl.	AURAMINES.		*e.g.*, Auramine O (B).
	Colourless, ethereal solution. Green fluorescence. Aqueous solution precipitated by KOH, hardly altered by HCl. Turns red with HNO_3.	ACRIDINES.		Phosphine.
	Colourless, or coloured ethereal solution. Non-fluorescent. Colour yielded to acetic acid — reddish-violet, blue, and green without fluorescence. Aqueous solution usually decolorized on warming with KOH, and coloured yellow by HCl (excepting fuchsin).	FUCHSINES (non-sulphonated).		Fuchsine.
	Ethereal solution colourless and non-fluorescent. Acetic acid coloured rose and fluoresces. Aqueous solution decolorized with KOH. — PYRONINES (coloured yellow by HCl. Direct for cotton wool).			Pyronine (G).
	— RHODAMINES (non-sulphonated. Unaltered by HCl.			Rhomamine S (By).
The coloured ethereal solution does not yield its colour to acetic acid. NEUTRAL COLOURING MATTERS. Insoluble in water. Soluble in alcohol.	The ethereal solution is yellow, and non fluorescent. Alcoholic solution yellow, non-fluorescent, and unaltered by aqueous acids and alkalies.	QUINONEPHTHALONES.		Quinoline yellow A (soluble in alcohol).
Ethereal solution colourless. Yields nothing to acetic acid. ACID COLOURING MATTERS. Soluble in water. Fixed on wool in acid bath (HCl). — The aqueous solution heated with fat-free wool to boiling. — Does not dye the wool.	Yellow colouring matters. No fluorescence in water. Unaltered by aqueous acids and alkalies.	SULPHONATED QUINONEPHTHALONES.		Quinoline yellow A (soluble in water).
	Reddish-violet, blue, or green colouring matters. Usually decolorized by KOH, little changed by HCl.	SULPHONATED FUCHSINES.		Fuchsine S (B).
	Red or violet colouring matters. Soluble in water with fluorescence. Precipitated by HCl. Changed but little, or not at all, by KOH.	SULPHONATED RHODAMINES.		Violamine R (M).
	Dyes the wool. — Brownish-yellow or orange colouring matters. Aqueous solution ± fluorescent. Fixed directly on silk, wool, and cotton.	THIAZOLES.		Primulin (B).

Class IV.—Colouring Matters not Reduced by $SnCl_2 + HCl$. Containing the Oxy-quinone Carbon Chromophore $O=R=C=$.

The alcoholic solution of the colouring matter, treated with a few drops of a dilute solution of Fe_2Cl_6 (1 : 1000).

Remains unaltered.

Non-amido Triphenyl-methane Colouring Matters usually soluble in water and direct for wool.

The colouring matter is dissolved or suspended in boiling water.

Not directly fixed on wool. Most of them insoluble in water. Soluble in alcohol without fluorescence.	Aurins.	$C(R^1)(R^1)(=R=O)$	Aurin.
Fixed directly on wool. Most of them soluble in water and alcohol. Fluorescence.	Phthaleïns.	$C(R^1)(R^1)(=R=O)$	Eosin.

Changes to green or olive-green.

Oxy-ketone Colouring Matters. Most of them insoluble in water, and indirect for fibres.

The original colouring matter is treated with faintly alkaline water (KOH. 1 per cent.).

Dissolves with yellow or reddish-yellow colour. — **Monoketones.**

The alkaline solution treated with excess of HCl.

Inclined to decolorization, especially on warming (with decomposition).	Benzophenones.	$CO(R)(R)$	Alizarin yellow A (B).
Coloured intense yellow without decomposition.	Flavones.	$CO(R)(O)(C=C)$	Quercetin.

Dissolves with red, reddish-violet, violet, green, or blue colour. — **Diketones (quinones).**

The alkaline solution acidified with acetic acid.

The free colouring matter precipitated. Usually soluble in ether, and indirect for fibres.	Non-Sulphonated Anthraquinones.	$CO(R)(R_1)CO$	Alizarin.
Colouring matter remains in solution. Insoluble in ether, fixed directly on wool.	Sulphonated Anthraquinones.		Sulphonated alizarin (alizarin-red).

II. *The Separation of Colouring Matters in a Mixture.*—It is sometimes possible to effect a separation by treating the mixture with water or alcohol at the ordinary temperature, or with the aid of heat; but as a rule extraction with ether or by fixing the dye on wool or other fibre is the most promising method. The behaviour of ether and wool is very similar, both extracting the free colouring matters, but not their salts. At the same time, not all colours soluble in ether can be fixed upon wool.

Extraction with Ether.—It is possible to separate basic from acid colouring matters by adding potash to a dilute aqueous solution of the dye and shaking with ether, when the free bases dissolve in the ether, leaving the acids in the aqueous layer. The details of the process are as follow: 100 c.c. of the aqueous solution of the colouring matter are decomposed with 1 c.c. of a 20 per cent. solution of potash, and shaken with three times the volume of ether. The aqueous alkaline solution of the acid colouring matter is neutralized with acetic acid and examined subsequently. The ethereal solution of the colour base is washed with faintly alkaline water, and then shaken with one-third of its volume of 5 per cent. acetic acid. The acid layer is separated, and on evaporation on the water-bath leaves the colouring matter as a

residue. In those cases in which the colour remains in the ether, the latter is evaporated. In the presence of potash some few acid or neutral colouring matters are also extracted by the ether, as, for example, quinoline yellow, indophenol blue (soluble in alcohol), the various sudans, etc. These are all insoluble in water, but soluble in alcohol.

In the extraction of the colour bases different alkalies have different liberating powers. Thus, safranine requires caustic potash, while for fuchsine ordinary ammonia is sufficient; others, again, such as the indulines, oxyazines and acridines, have their colour bases set free by very dilute ammonia; and others, such as chrysoidine, Bismarck brown, rhodamine S(By), Victoria blue, etc., dissociate in dilute aqueous solution. Thus, separation can often be made by successively shaking the aqueous solution with ether, first alone, then together with 1 per cent. ammonia, then with concentrated ammonia, and finally with 20 per cent. potash. A further separation of the bases taken up by the ether can sometimes be effected by shaking the ethereal solution with an equal volume of water, some being taken up by the water, others remaining in the ether. In this way it is possible to separate acridine yellow from the very similar phosphine. The colour bases remaining in the ether differ in their behaviour towards 5 per cent. acetic acid, some combining with it, others remaining unaltered.

The acid colouring matters not extracted by ether from an alkaline aqueous solution can be separated by methods similar to those used with the colour bases. By successive extractions with ether, they can be separated into three groups: (1) those extracted by ether in the presence of acetic acid of 1 per cent. strength; (2) those soluble in ether in the presence of hydrochloric or sulphuric acid; and (3) those insoluble in ether. Erythrosin can thus be separated from roccellin and from Bordeaux B, and direct yellow (A) from Congo brown R (A) and from Congo red (A). By treating the ethereal solutions with water and dilute ammonia, as in the case of the basic colouring matters, a further separation can often be made, as, for instance, picric acid from Martius yellow.

Separation by means of Wool.—When a separation cannot be made with ether, it is often possible by means of wool. An aqueous solution of the colouring matter (1 : 1,000) is rendered faintly alkaline by the addition of four or five drops of ammonia per 100 c.c., some wool added, and the liquid heated to boiling with constant stirring. This is repeated with fresh supplies of wool so long as the fibres are dyed. The wool is washed with boiling ammoniacal water, then with pure water, and extracted with 5 per cent. hot acetic acid. On evaporating this extract on the water-bath, the basic colouring matters are left, and can then be further separated.

The use of wool is more effective in the separation of acid colouring matters, some of which are directly fixed by it. A 0·1 per cent. solution of the colour mixture is acidified with hydrochloric acid (three to four drops per 100 c.c.), brought to the boil, and wool immersed for from three to five minutes with continual stirring. This treatment is repeated as with the basic colours. The dyed wool is washed with acidified water, and then with pure water, and the colour extracted with 5 per cent. ammonia. By boiling the liquid until the ammonia is expelled, the direct colours are obtained in neutral solution. Since some of the indirect colours are taken up to

a slight extent by the wool fibre, this solution should also be treated with the wool in order to effect complete separation. In this way the following colours can be separated from each other:

Direct. { Bordeaux B (A). { Biebrich scarlet. { Acid yellow (A).
Indirect. { Œnocyanin. { Cochineal. { Saffron.

The direct colours have a great difference in their affinity for wool, and with some it is dyed readily in a strongly acid solution (*e.g.*, those with oxysulpho- groups), while others, of both acid and basic character, also dye in a neutral solution. The following colouring matters can thus be separated:

Fixed in neutral bath. { Alkali violet (B). { Acid violet 4BN. { Orseille
,, acid ,, { Ponceau 6RB (A). { New coccine (A). { Bordeaux B (M).

By using a strongly acid solution (1 c.c. HCl to 200 c.c.) the following separations can be made:

Fixed in strongly acid bath. { Bordeaux S (A). { Bordeaux B (A).
,, slightly ,, { Orange G (A). { Methyl orange.

When a separation of the constituents of a mixture cannot be effected by means of wool, cotton-wool must be tried. It has the property of fixing some of the direct dyes for wool, leaving others in the bath. Thus:

Direct for cotton-wool. { Carbazol (B). { Cotton yellow R (B).
Indirect ,, ,, { Diamond yellow R (By). { Phloxin B (B).

With cotton also some of the dyes are more readily fixed than others, especially under varying conditions of the acidity and concentration of the bath. Thus, in slightly acid bath (HCl) brilliant Congo (A) is readily fixed, but brilliant yellow (A) only with difficulty.

If none of these means have effected a separation, other solvents, such as petroleum spirit, amylic alcohol, chloroform, etc., should be tried. With petroleum spirit, for example, eosin can be separated from Martius yellow. C. A. M.

Use of Basic Lead Acetate in the Polarimetry of Sugar Solutions. Prinsen-Geerligs. (*D. Zuckerind*, 1898, xxiii., 1753; through *Chem. Zeit. Rep.*, 1898, 320.)—Although basic lead acetate does not precipitate pure lævulose, yet from impure solutions, and notably such as contain much saline matter, it carries down more or less sugar with it. On preparing artificial liquids from honey and common salt to imitate natural juices (13·44 per cent. of invert sugar and 7·68 per cent. of sodium chloride), and adding increasing proportions of basic lead acetate, the amount of lævulose precipitated varied between 3·43 and 23·14 per cent., thus causing a corresponding increment in the dextro-rotatory power of the solution. When sufficient of the reagent is introduced to decompose all the salts present in the juice, further additions have no effect on the sugar. As lead nitrate, normal acetate, bone-charcoal, and zinc dust are all unsatisfactory, it is necessary to employ basic acetate; and in order to obtain comparable results, the author suggests that the quantity of lead required to render molasses fit for optical examination shall be determined, and the same quantity used for all other kinds of syrup and crude sugars. The precipitated

lævulose also contains glucose, but the proportion of the latter never exceeds 25 per cent. of the total sugar thrown down. F. H. L.

The Estimation of Indigo on Fabrics. A. Binz and F. Rung. (*Zeit. angew. Chem.*, 1898, 904-905.)—Renard (*Bull. Soc. Chim.*, xlvii., 41, 1887) proposes to heat a weighed portion of the fabric with a measured quantity of hydrosulphite solution, and determine the indigo white in an aliquot portion of this. His method is objectionable, since indigo white is obstinately retained by the tissue. The method of extracting the fabric with aniline in a Soxhlet apparatus, as proposed by Hönig (*Zeit. ang. Chem.*, 1889, 280), is much simpler. As, however, boiling aniline exercises a destructive action on indigotin, glacial acetic acid, as proposed by Brylinski (*Rev. génér. mat. color.*, 1898, 52), forms a better solvent.

The paper contains an account of the authors' comparative experiments with Brylinski's method slightly altered, and with a modification of the hydrosulphite method. In the latter the fabric was treated with the hydrosulphite solution on the water-bath until completely decolorized, and the indigo-white removed from the tissue by repeated washing out with hot water, of which from 2·5 to 3 litres were required to remove only a few decigrammes of the dye. The whole of the solution of indigo white thus obtained was oxidized by means of a current of air, and the indigo collected on a tared filter-paper, dried and weighed.

In the experiments on the glacial acetic acid method a suitable quantity (10 grammes) of dyed cotton was heated for three or four hours over a naked flame with 150 c.c. of glacial acetic acid, and then poured into 300 c.c. of water. Instead of now filtering off with a suction-pump, as Brylinski directs, the authors found it preferable to add 150 c.c. of ether. The whole of the indigotin remained in suspension in the ethereal layer, and this was separated from the diluted acetic acid by means of a separatory funnel. The former was filtered off, the precipitate washed with alcohol and ether, and dried at 110° C.

The results obtained by the acetic acid method were somewhat higher than those of the hydrosulphite process, and this the authors considered might be due to over-reduction by the hydrosulphite, or to an acetyl derivative of cellulose dissolving in the acetic acid. Brylinski states that the latter body is soluble in ether, and the authors found that this was the case; for, on extracting two equal weights of a dyed fabric, to one of which about forty times its weight of bleached cotton-wool had been added, the amounts of indigotin obtained were practically the same. C. A. M.

The Determination of Sulphur in Asphalt. E. H. Hodgson. (*Jour. Amer. Chem. Soc.*, vol. xx. [11], pp. 882-889.)—Various samples were examined by the methods of Carius (heating with strong nitric acid in sealed tubes), Peckham (deflagration), Eschka (ignition with calcined magnesia), and by fusion with sodium peroxide. From the results the sealed tube method appears the most accurate, but is a lengthy operation, and the tubes are liable to explode. Of the others, that of Eschka is probably the best, giving good results and requiring least time and attention.

C. S.

Use of Persulphates to Detect Albumin in Urine. C. Strzyowski. (*Schweiz. Wochs. Chem.*, 1898 [48]; through *Deutsche Chem. Zeit.*, 1899, xiv., 2.)—A 10 per cent. aqueous solution of potassium, sodium, or preferably ammonium persulphate, precipitates albumin from either acid or alkaline urine, and does not throw down peptones or urates. By means of a pipette the reagent is brought underneath a layer of the suspected urine contained in a test-tube, when even in dilutions of 1 : 100,000 a grayish-white turbidity is produced at the line of contact. In presence of bile pigments the colour is bright green. F. H. L.

Estimation of Chlorides in Urine, Wine, Beer, and Cider. Loubiou. (*Rep. Pharm.*, 1898 [3], x., 493; through *Chem. Zeit. Rep.*, 1898, 319.)—Lead peroxide oxidizes in the cold those constituents of urine which act on silver nitrate, and decolorizes it sufficiently to enable potassium chromate to be used as an indicator in the titration of chlorides. 20 c.c. are shaken with 2 or 3 grammes of the peroxide and filtered; 10 c.c. of the filtrate are mixed with 5 drops of saturated potassium chromate, diluted to 50 c.c., and titrated as usual. Similarly, employing Blarez's process, 50 c.c. of white or red wine (beer, cider, or vinegar) are treated with 3 c.c. of chromate, 50 grammes of salt, 100 c.c. of water, and 5 grammes of lead peroxide; the whole is shaken, filtered, and an aliquot portion titrated. F. H. L.

Separation of Albumoses from Peptones. P. Muller. (*Zeits. Physiol. Chem.*, 1898, xxvi., 48; through *Chem. Zeit. Rep.*, 1898, 320.)—After experiments with the salts of various heavy metals, and especially with uranium acetate, the author prefers ferric hydrate for this purpose. The liquid to be treated is mixed with an equal volume of 30 per cent. ferric chloride solution, and alkali is added till it is only just acid. The precipitate is removed, two or three pinches of zinc carbonate are thrown into the filtrate, which is shaken thoroughly and again filtered. It will be clear, colourless, and free from albumoses, giving no turbidity (or only a very slight one visible against a dark background) when saturated with ammonium sulphate. The method has proved successful in all cases where it was tried except with Witte's peptone, which involved a concentration of the filtrate to one-fourth or one-fifth of its volume, followed by a second treatment with a few drops of ferric chloride and zinc carbonate as before. F. H. L.

Sicilian Sumach and its Adulterants. F. Andreasch. (*Gerber*, 1898; through *Zeits. angew. Chem.*, 1898, 1154.)—True sumach is the product of *Rhus coriaria*, an Arabian plant; but it is blended with the leaves of the Sommacco fimeneddu, which contain less tannin. Inferior specimens are mixed with stalk, earth, sand, and already extracted sumach. As adulterants there are added leaves of the carobbe, of *Cistus salvifolius* (very frequently in Sicily), of the fig, vine, of *Ailanthus glandulosa*, *Pistacia lentiscus* (Stinco sondro, Lentisco), and of *Tamarix Africana* (Bruco, albero di Giuda): the two latter being the most common. The amount of tannin and non-tannin in sumach and its chief adulterants is shown in the annexed table. The average proportion of tannin in pure sumach may be taken at 23 or 24, seldom falling

below 22: a yield of under 20 per cent. points either to the presence of other parts of the plant or to sophistication. Large additions of stalk, etc., can usually be detected by the eye, if not by examination of the aqueous extract; for the bulk of the red colouring matter in the woody fibre passes into solution, and on acidification with acetic acid yields a weak but distinct red tint.

	Tannins.	Non-tannins.
Silician sumach ...	21–27·5 per cent. ...	16–22 per cent.
Pistacia	13–17 ,, ...	20–26·6 ,,
Tamarix	8·3–9 7 ,, ...	23–26·5 ,,
Ailanthus	10 ,, ...	17·5 ,,

When a little formaldehyde is dropped into a neutral decoction of pistacia, a pale-yellow precipitate is produced. Even if the material is chiefly genuine sumach, a yellowish-coloured cloud forms, which appears as a precipitate in time, but only settles after several days. The deposit is gelatinous and coheres on the filter, so that it can scarcely be washed. The substance is insoluble in cold water; but it does not represent a quantitative separation of the pistacia. If the test be carried out on very old samples of sumach, which perhaps have fermented, or if much tamarix be present, the formalin may produce a precipitate even in the absence of pistacia; but the latter cannot be mistaken for the real deposit: it is only small in amount, never gelatinous, and settles in twelve hours.

When solid potassium cyanide, or its strong solution, is stirred up with a decoction of tamarix, or sumach adulterated with tamarix, a flocculent dark-yellow precipitate is formed which settles rapidly. Pure sumach gives no deposit or a mere trace, and the reaction is characteristic of tamarix alone among the possible adulterants. The reagents in both tests must be pure, and the solution examined should be perfectly clear, and must not exceed industrial strength, viz., about 0·75 gramme of tannin per 100 c.c. F. H. L.

Estimation of Oil of Bergamot. A. Soldaini and C. Berté. (*Boll. chim. farm.*, 1898, xxxvii., 577; through *Chem. Zeit. Rep.*, 1898, 311.)—The usual adulterants added to bergamot oil are turpentine and (or) lemon oil. To detect them, 15 c.c. of the sample should be distilled at a pressure of 20 or 30 mm. till 5 c.c. have passed over, and the opticity of the oil, the distillate, and the residue determined. The annexed table indicates the influence of both or either of the impurities mentioned:

	Temperature.	Oil.	Residue.	Distillate.
Pure bergamot oil	14° C.	+14° 50′	−0° 56′	+41° 20′
+5% of turpentine	14° C.	+12° 36′	−0°	+35° 28′
+5% of lemon oil	14° C.	+17° 11′	+3° 20′	+42° 28′
+2·5% of turpentine and 2·5% of lemon oil	13·5° C.	+14° 55′	+2° 40′	+40° 20′

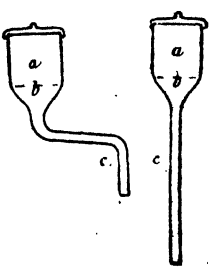
F. H. L.

INORGANIC ANALYSIS.

Bacterial Detection of Arsenic. F. Abba. (*Centralbl. Bakteriol.*, 1898, [2], iv., 806; through *Chem. Zeit. Rep.*, 1898, 310.)—Gosio has already shown that when *Penicillium brevicaule* is cultivated in presence of arsenic a garlic-like odour is produced. The present author finds that this process is not only simpler than chemical methods of detecting arsenic, but, after investigating fifty other substances, that it is quite characteristic. It is also more delicate, for a piece of Indian fur 1 square millimetre in area gave the reaction, whereas even 5 square centimetres did not show the Marsh test. The material to be examined is placed in sliced potato, sterilized at 115° for twenty minutes, inoculated with 0·5 c.c. of sterilized water containing spores of the *P. brevicaule* in suspension, and cultivated either at 37° or at ordinary temperatures. Fresh cultures of the penicillium need only be prepared once a year. F. H. L.

Colorimetric Estimation of Iron in Water. A. Seyda. (*Chem. Zeit.*, 1898, xxii., 1086.)—Lunge's colorimetric process for determining traces of iron in aluminium sulphate is very suitable for the examination of water. Instead of matching the colour of the aqueous solutions as in the usual method, each is shaken up with a measured quantity of ether, and after the layers of ether have been adjusted to the same length, the colour comparison is made in these. If the water under examination contains much organic matter an error may be introduced; in this case the samples must first be evaporated in platinum, ignited, fused with sodium carbonate, and the melt dissolved in hydrochloric acid. F. H. L.

Remarks on the Determination of Zinc and Manganese as Sulphide. E. Murmann. (*Monatshefte für Chemie*, xix., 404.)—On account of the difficulty of filtration, the determination of zinc or of manganese as sulphide is rarely resorted to, though the method is capable of yielding very accurate results. The author overcomes this drawback by adding mercuric chloride to the solution before precipitation. The precipitated mercuric sulphide enables the finely divided zinc or manganese sulphide to be filtered off and washed without difficulty, whilst in the subsequent ignition of the precipitate the mercuric sulphide is completely expelled. The author has also devised a new form of crucible specially adapted for the ignition of such precipitates in a current of hydrogen gas. Its construction is shown in the accompanying figure. The crucible *a* has a perforated bottom *b*, to which is attached a tube *c*, which can be either straight or bent at right angles. A perforated plate fits into the bottom, and between the perforated bottom and the perforated plate is a plug of asbestos as used in the Gooch crucible. The mouth of the crucible is covered by an ordinary crucible lid. The precipitate is filtered off and also dried and ignited in the crucible, whilst by means of the tube at the bottom a current of hydrogen can be passed through it during the ignition. The crucible can, of course, be used for other similar purposes. H. H. B. S.



Estimation of Manganese as Phosphate. E. A. Gooch and M. Austin. (*Zeits. anorg. Chem.*, 1898, xviii., 339.)—In this process the metal is thrown down as trimanganese orthophosphate, the precipitate converted into ammonium manganese orthophosphate, and finally weighed as manganese pyrophosphate. Its accuracy, therefore, depends upon the rapidity and completeness with which the $Mn_3P_2O_8$ is changed into $MnNH_4PO_4$, and the degree of insolubility of the latter, which, as Fresenius and Munroe have shown, is considerably affected by the presence of ammonium chloride. The present authors have investigated the method under various conditions, ultimately finding that a very large excess of ammonium chloride (200 molecules of NH_4Cl to 1 of $MnNH_4PO_4$ in 100 or 200 c.c. of liquid) is absolutely indispensable to a quantitative conversion of the original precipitate into the double phosphate, and, moreover, that $MnNH_4PO_4$ is not appreciably dissolved by ammonium chloride solution, provided excess of the soluble phosphate employed as a precipitant is always present.

Details for the successful operation of the process are as follows: The original faintly acid liquid (containing not more than 0·2 gramme of metallic manganese) is mixed either in a platinum or a glass vessel with 20 grammes of solid ammonium chloride, 5 or 10 c.c. of a cold-saturated solution of microcosmic salt, and diluted to 200 c.c.; dilute ammonia is then added in the cold drop by drop until it forms a slight excess. The whole is heated till the precipitate becomes crystalline, allowed to cool for half an hour, filtered quickly through a Gooch crucible with the aid of a pump, and washed (preferably) with very weak ammonia. The filtrate is always quite free from manganese, and if the precipitate retains traces of ammonium chloride, this can only lead to the formation of equally minute quantities of manganese chloride. It is better to arrange for a precipitate weighing about 0·4 gramme; but even with smaller amounts the final error will average not more than 1 mgm. of manganese.

F. H. L.

The Determination of Chromium Oxide in Algerian Phosphates. G. Schüler. (*Zeits. angew. Chem.*, 1898, 1101-1103.)—With the exception of Shephard's chromium phosphate ($Cr_2P_2O_8$) chromium has hitherto only been found in traces in phosphates, and never, to the author's knowledge, in calcium phosphates. In Algerian phosphates, however, which have recently become of considerable importance in commerce, chromium appears to be a constant though small constituent.

For the quantitative estimation of the chromium the author employs a modification of J. Clarke's method of oxidation with sodium peroxide (*Journ. Chem. Soc.*, lxiii., 1079). Ten grammes of the phosphate are dissolved in the usual mixture of hydrochloric and nitric acids, and the filtrate and washings evaporated to a pasty consistency, in order to expel the excess of acid. The moist residue is taken up in as small a quantity as possible of hydrochloric acid, oxidized by boiling with sodium peroxide, and the residue again treated in the same manner. The united filtrates and washings (containing the chromium as sodium chromate) are boiled down, and in order to completely remove the excess of sodium peroxide are again strongly diluted and evaporated. The liquid is then acidified, and the chromic acid titrated in the usual manner.

In this way the author found 0·057 per cent. of chromic oxide in a large representative sample of Algerian phosphate. C. A. M.

Volumetric Estimation of Molybdenum. **F. A. Gooch and J. T. Norton.** (*Zeits. anorg. Chem.*, 1898, xviii., 312.)—The iodometric method of estimating molybdenum outlined by Friedheim and Euler (ANALYST, 1896, xxi., 25) suffers from the defect that the precautions taken to avoid the disturbing influence of atmospheric oxygen are by no means sufficient. To be fairly accurate, the process requires carrying out as follows: The amount of molybdate taken for analysis must not contain more than 0·3 gramme of MoO_3; the excess of potassium iodide over that decomposed by the molybdate must not be greater than 0·5 gramme; the liquid should consist of 20 c.c. of the strongest hydrochloric acid and 20 c.c. of water; it must be boiled down till only 25 c.c. are left in the flask; and the distillation must be conducted in a current of carbon dioxide carefully freed from air. Even under these conditions an error, corresponding to some 0·3 milligramme of molybdenum trioxide, is introduced by the oxygen dissolved in the water and acid; to obtain the best results, therefore, these reagents should be boiled before use.

As an alternative way of determining molybdenum, the molybdate may be reduced to pentoxide by heating it with acid and an iodide in an Erlenmeyer flask lightly closed with a bulb-tube, boiling off the excess of iodine, making the liquid alkaline, and finally titrating it with standard iodine solution. F. H. L.

Note on Drown's Method of Determining Silicon in Steel. **G. Auchy.** (*Jour. Amer. Chem. Soc.*, 1898, xx., 547-549.)—There is some reason to believe that in determining the silicon in steel by Drown's method some of it escapes dehydration owing to the fact that, as the sulphuric acid becomes more concentrated, part of the iron salts separate out and form a protecting envelope round the gelatinous silica.

To obviate this the author suggests evaporating with *aqua regia* and sulphuric acid, instead of with nitric and sulphuric acids, the liquid being rapidly boiled down in a small porcelain dish on a very hot plate, with a watch glass suspended over the basin by means of glass rods. In this way the iron salt is kept in solution until the very last moment, when it suddenly crystallizes out and fumes of sulphuric acid appear. If a second similar evaporation be made before filtering, it is hardly likely that any appreciable quantity of silica will escape dehydration.

A number of test analyses were made in this way with steels containing 0·25 per cent. of silicon, and not more than an additional 0·005 per cent. was obtained by the second evaporation. A steel which gave 0·122 per cent. with one evaporation gave 0·126 per cent. on repeating the analysis and evaporating twice before filtering.

It is also stated that the evaporation proceeds quietly and without bumping or spurting, even at the moment of solidification and after, so that no attention is required. C. A. M.

APPARATUS.

A New Form of Water Blast. (*Amer. Chem. Journ.*, 1898, xx. 577-580.)—The essential parts of this apparatus are: (1) A receiver *A* in the form of a cylinder (8 inches long and 6 inches in diameter) divided into three compartments *b*, *c*, and *d* by the partitions *p* and *p'*. Between *b* and *c* is a circular opening 2 inches in diameter, and two smaller openings are placed between *c* and *d*. (2) An injector tube, *l*, 7½ inches long and ⅝ inch outside diameter, provided at the top with the funnel-shaped projection *g*. (3) An outlet tube *f* with a diameter equal to that of *l*, extending upward from *c* and curving over and downward to *g*. The tube *t* ($\frac{7}{16}$ inch in diameter) opens into *f* and extends to *g*. *S* is a stopcock opening into *d*, and *K* a nozzle with an orifice $\frac{1}{16}$ inch in diameter attached to the upper part of *f*. The axes of *K* and *l* are made to *exactly* coincide.

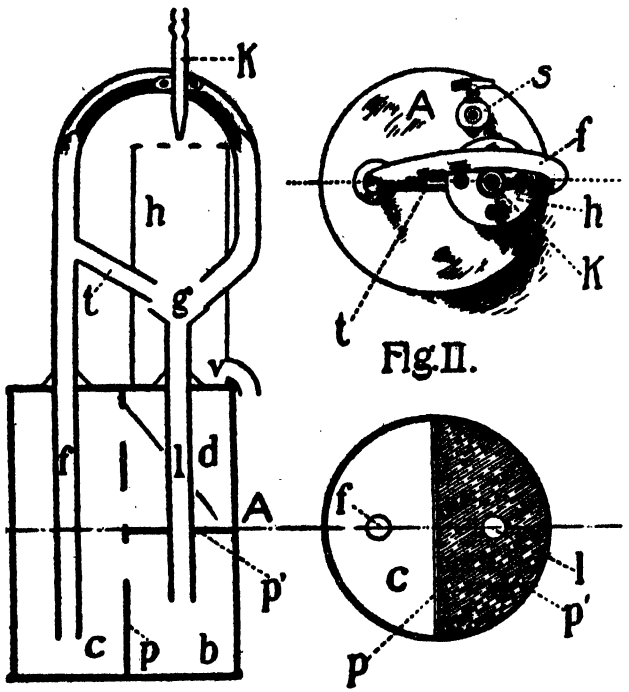


Fig. I.

Fig. II.

Fig. III.

In action the cock *S* is opened, and the water turned on. The jet of water issuing from *K* passes through *l* and falls into the compartment *b*. When the level of the water in *b* rises above the lower end of the tube *l*, a considerable volume of air is swept into *A*, escaping through *f* and *S*, and later through *S* only. The water flows from *b* into *c* through the opening in the partition. The cock *S* is now closed, and the increase of pressure in the receiver forces the water up through *f* and into *g*. The jet from *K* now strikes on the surface of the water in *g* and drives it down into *A*, together with a large volume of air. If the cock *S* be now opened a current of air of sufficient pressure to supply a large blast lamp will be obtained. Spurting is prevented by surrounding *g* with a cylindrical case *h*, having openings in the top to allow air to enter, and an overflow pipe *v* for the escape of water.

Fig. 1 is a vertical section of the complete apparatus; Fig. 2 is a projection from above; and Fig. 3 a section through the middle of the receiver.

The advantages claimed for this blast over that of Richards and that of Muenke are: That the orifice for the entrance of air and water is of such ample size that any

considerable friction is avoided; that owing to the injector tube being perfectly straight the jet of water is not deflected, and the maximum effect is obtained; and that the energy is transmitted to a much greater volume of water, which circulates continuously from *f* through *l* and acts as a piston which completely fills the cross section of the tube.

This apparatus can be obtained from Eimer and Amend, New York. C. A. M.

Apparatus for Increasing the Speed of Filtrations. H. von Winkler. (*Chem. Zeit.*, 1898, xxii., 1066.)—This apparatus is specially suitable for the filtration of minute quantities of liquid where the use of the filter pump is impracticable. It consists essentially of a glass plate, one side ground true, having at its centre a tube projecting at right angles to which a rubber tube can be affixed. A funnel with a ground edge is carefully fitted with a strong paper and filled with the liquid; the disc is placed on top and held in position with the fingers, while air is blown through the tube by means of the mouth, thus increasing the effective pressure. The disc may be of vulcanite, with a soft bearing surface if preferred; or the glass may be moistened (in order to make a better joint) with some of the same liquid as that under treatment. F. H. L.

REVIEW.

AN ATLAS OF BACTERIOLOGY. By CHARLES SLATER, M.A., M.B., M.R.C.S. (Eng.), F.C.S.; and EDMUND J. SPITTA, L.R.C.P. (Lond.), M.R.C.S. (Eng.), F.R.A.S. (London: The Scientific Press, Limited.) Price 7s. 6d.

This work consists of 111 original photomicrographs, together with explanatory text, a photographic introduction, and a bacteriological introduction. The photographic introduction will be found very useful to anyone who desires to keep a permanent record of his bacteriological results, containing as it does many practical hints on the selection of objectives and general optical apparatus. The results obtained by the authors show evidence of much patience and manipulative skill, while the process reproduction is excellent. In the bacteriological introduction a description is given of the morphology and reproduction of the *Schizophytes*. The photographic illustrations of cultures and microscopic appearances of the more important bacteria follow, together with a résumé of the chief characteristics of each. Amongst other organisms, *Bacillus anthracis*, *B. tuberculosis*, *B. typhosus*, *B. coli communis*, *B. diphtheriæ*, *Spirillum choleræ Asiaticæ* are very fully illustrated. Attention is to be directed to plates 16 (*B. anthracis*, envelope), 17 (*B. anthracis*, spore formation), 18 (*B. anthracis*, stab culture), 53 (*B. typhosus*, gelatine streak culture), 60 (*B. coli communis*, gelatine streak culture), 69 and 70 (*B. diphtheriæ*, culture), and the plates illustrating the differentiation of *Sp. choleræ Asiaticæ* (79 and 80), and *Sp. Finklerii* (84 and 85). In a future edition it may be hoped that illustrations of colonies of many of the more important pathogenic and non-pathogenic organisms, as they appear when grown on phenolized agar jelly at 37° C., will be added, for although they lose many characteristics, yet it is in this form that they are generally met with in the bacteriological examination—for instance, of water. The appearance of actual colonies and characteristic growths

would seem to be of equal importance with the microscopical appearance of the organisms themselves. As it is, the book is one which every analyst should possess for reference who systematically examines water bacteriologically. J. F. H. G.

INSTITUTE OF CHEMISTRY OF GREAT BRITAIN AND IRELAND.

THE following is a list of the names of the candidates who passed the examination of the Institute of Chemistry held in January, 1899:

PRACTICAL EXAMINATION FOR THE ASSOCIATESHIP (under regulations in force prior to October 1, 1893).—Chapman, Arthur Jenner, Finsbury Technical College, London; and with Messrs. F. and A. C. Claudet, FF.I.C. Forster, Ferdinand Edward Paul, Finsbury Technical College; registered student under Messrs. F. E. Lott, F.I.C., and C. G. Matthews, F.I.C. Lishman, George Percy, B.Sc. (Edin.), Surgeons' Hall, Edinburgh; registered student under Professor W. Ivison Macadam, F.I.C.

INTERMEDIATE EXAMINATION (under new regulations).—Aston, Stafford, University College, London. Blackman, Walter Lionel, Royal College of Science, London, and St. Bartholomew's Hospital. Coysh, Basil Radcliffe, King's College, London. Hewitt, Henry Dixon, Finsbury Technical College; registered student under A. C. Chapman, Esq., F.I.C. Hill, Alfred, University College, Nottingham. Kinnersley, Henry Wulff, Merchant Venturers' Technical College, Bristol, under Professor Julius Wertheimer, B.A., B.Sc., F.I.C.; registered student under Dr. E. H. Cook, F.I.C.; and at King's College, London. Rogers, John, Glasgow and West of Scotland Technical College. Shedden, Frank, B.Sc. (Lond.), Mason University College, Birmingham. Wright, Leonard Victor, B.A. (Cantab.), Sidney College, Cambridge.

FINAL EXAMINATION FOR THE ASSOCIATESHIP (under new regulations). — *In Branch A (Mineral Chemistry)*: Golding, John,* School of the Pharmaceutical Society; Laboratory of the Royal Agricultural Society of England; University College, Nottingham. Hancock, Walter Charles,* B.A. (Cantab.), Caius College, Cambridge; with Messrs. C. F. Cross, F.I.C., and E. J. Bevan, F.I.C.; and at King's College, London. Marshall, Arthur, A.C.G.I., City and Guilds of London Institute. *In Branch B (Metallurgical Chemistry)*: Imrie, John, Glasgow and West of Scotland Technical College. Wild, Roland Cecil, King's College, London; registered student under C. J. Head, Esq., F.I.C. *In Branch D (Organic Chemistry)*: Gilles, William Setten, A.C.G.I., City and Guilds of London Institute. Hinks, Percy John, A.R.C.Sc. (Lond.), Royal College of Science, London. Joyce, Thomas Goode, B.Sc. (Lond.), Mason University College, Birmingham. *In Branch E (the Analysis of Food and Drugs and of Water)*: Gilbard, John Francis Hutchins,* Finsbury Technical College; and with Dr. Bernard Dyer, F.I.C. Hackman, Charles Adolphus, King's College, London; and with A. C. Chapman, Esq., F.I.C. Young, Francis Samuel, M.A. (Oxon.), Queen's College, Oxford, and Caius College, Cambridge.

The examiners were Professor Percy F. Frankland, F.R.S., F.I.C., and Otto Hehner, Esq., F.I.C.

* For the Fellowship.

THE ANALYST.

MARCH, 1899.

PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS.

THE annual general meeting of the Society was held on Wednesday, February 1, in the Rooms of the Chemical Society, Burlington House, the President (Dr. Bernard Dyer) occupying the chair.

The minutes of the extraordinary general meeting and of the ordinary general meeting, held on January 4, were read and confirmed.

Certificates of proposal for election to membership in favour of the following candidates were read for the first time: Percy T. Adams, assistant to Dr. Matthew A. Adams, Maidstone; Arthur French Angell, assistant to Mr. Arthur Angell, Southampton; John B. Ashworth, assistant to Mr. Alfred Smetham, Liverpool; Alfred Joseph Bull, assistant to Mr. Wm. Chattaway, and senior assistant in the Physical Laboratory, Birkbeck Institution, London; Charles Crocker, assistant to Messrs. Morgan, Son and Seyler, Swansea; John S. Ford, analytical chemist, Edinburgh; C. A. Hackman, A.I.C., assistant to Mr. Alfred C. Chapman, London; Norman Leonard, B.Sc., F.I.C., assistant to Dr. Stevenson, Guy's Hospital, London; J. F. Liverseege, F.I.C., assistant analyst to the Corporation of Birmingham; Richard Murray, chemist to Messrs. Brotherton and Co., Leeds; G. E. Scott-Smith, F.I.C., assistant to Mr. Alfred H. Allen, Sheffield; W. P. Skertchly, assistant to Mr. Otto Hehner, London; Harry Metcalfe Smith, F.I.C., assistant to Dr. Stevenson, Guy's Hospital, London; Lionel W. Stansell, assistant to Dr. Matthew A. Adams, Maidstone; and W. Northfield Yarrow, assistant to Mr. A. W. Stokes, London. All of these candidates were former associates of the Society, applying for election to membership under the revised rules recently adopted.

Mr. F. C. J. Bird and Mr. J. H. Heywood were elected members of the Society.

The HON. TREASURER (Mr. E. W. Voelcker) presented his report for the past year. He stated that the Society had a balance of £40 5s. 5d. standing to its credit, as against £32 12s. 8d. shown in the balance-sheet for 1897. The items most calling for attention were those classed as "General Expenses," and relating to postages, petty disbursements, etc., and the printing and stationery account. It would be seen, from the copies of the accounts which had been placed in the hands of the meeting, that these items showed a considerable increase as compared with the previous year. During 1898, however, the Council had considered it desirable to circulate among Members of Parliament, and among County Councils and other local authorities, a

report of the Council of the Society of Public Analysts dealing with the Sale of Food and Drugs Bill introduced by the President of the Local Government Board at the close of the session of 1897. This had involved a considerable outlay in printing, and a very heavy expenditure on postage. The securities of the Society on December 31 were the same as in the previous year; but since that date he had been able to make a further investment on behalf of the Society, so that its invested funds were now increased by £105. He was pleased to be able to announce that only one subscription remained outstanding.

A vote of thanks to the Hon. Treasurer and Auditors was proposed by Mr. CHAPMAN, seconded by Dr. SCHIDROWITZ, and carried unanimously.

The President then delivered his retiring address.

Mr. ALFRED H. ALLEN proposed a vote of thanks to the President for his address.

Mr. HEHNER seconded the proposition, which was put to the meeting by Mr. Allen and carried unanimously.

The PRESIDENT, having responded, moved from the chair that a vote of thanks should be passed to the President and Council of the Chemical Society for allowing the Society of Public Analysts the use of their rooms during the past year. The motion was seconded by Mr. CHATTAWAY and carried unanimously.

The PRESIDENT announced that the officers and Council for 1899 had been elected in accordance with the nominations made by the Council at the December meeting of the Society. He then vacated the chair in favour of the newly-elected President, Mr. W. W. Fisher, M.A.

Mr. FISHER, having assumed the chair, expressed his appreciation of the honour which the Society had done him in electing him to occupy that position, and the proceedings then terminated.

The following is a list of the officers and Council of the Society for 1899:

President.—W. W. Fisher, M.A.

Past-Presidents.—M. A. Adams, F.R.C.S.; A. H. Allen; Sir Charles A. Cameron, M.D., F.R.C.S.; A. Dupré, Ph.D., F.R.S.; Bernard Dyer, D.Sc.; Otto Hehner; Alfred Hill, M.D., F.R.S.E.; J. Muter, M.A., Ph.D., F.R.S.E.; Thomas Stevenson, M.D., F.R.C.P.

Vice-Presidents.—Charles E. Cassal; E. W. T. Jones; S. Rideal, D.Sc.

Hon. Treasurer.—E. W. Voelcker, A.R.S.M.

Hon. Secretaries.—E. J. Bevan, Alfred C. Chapman.

Other Members of Council.—Bertram Blount; Sidney Harvey; Arthur R. Ling; C. A. Mitchell, B.A.; C. G. Moor, M.A.; James Nimmo; Thomas A. Pooley, B.Sc.; F. Wallis Stoddart; R. T. Thomson; John White; Rowland Williams; W. C. Young.

ADDRESS OF THE RETIRING PRESIDENT.

(Delivered at the Meeting, February 1st, 1899.

In addressing you at the close of 1897, I had the melancholy duty of recording the deaths of an illustrious honorary member of the Society and of three ordinary members. This year, I regret to say, we have again to record the death of an illustrious honorary member, Lord Playfair, whose chemical career is sufficiently well known to chemists to render it unnecessary for me now to do more than call attention to the fact that it was in recognition of his interest in the question of food adulteration, and of his successful exertions in the House of Commons for the improvement of the Bill passed in 1875, that he was elected an honorary member of this Society. During the past year also we have again lost by death three ordinary members. One was our late lamented Vice-President, John A. R. Newlands, a chemist of remarkable ability, whose name will long live in connection with the history of the development of chemical thought during our time, on account of his early enunciation of what has since become known as the Periodic Law of Newlands and Mendelejeff. He was a very old and universally beloved member of this Society, and the rooms of the Chemical Society and other haunts of London chemists have been sadder and duller during the present session by reason of the disappearance from among us of his bright face and genial voice. Mr. J. Napier and Mr. F. P. Perkins, whom we have also lost during the year, were both old members of the Society, though they had not been in the habit for many years of attending its meetings. Mr. Napier was well known as Public Analyst for the borough of Ipswich and for the county of Suffolk, while Mr. Perkins had for many years occupied the position of Public Analyst for the city of Exeter.

Seven members of the Society resigned their membership during the year, and four associates were elected to the full membership of the Society. In addition to these, we elected five other new members and five associates. The net consequence of these changes is that we numerically lost one honorary member and one ordinary member, gaining one associate. The following figures show the numerical condition of the Society for the last four years:

	January, 1896.	January, 1897.	January, 1898.	January, 1899.
Honorary members ...	10	11	11	10
Members	210	218	223	222
Associates	26	29	31	32
	246	258	265	264

This is perhaps a fitting place for congratulating the Society upon having recently adopted, on the advice of the Council, a considerably modified scheme of constitution and rules. The changes which have been made had been for a long time under the consideration of the Council and officers of the Society, and I hope and believe that they will result in a gain on the part of the Society, in efficiency as well as in numerical strength.

It is scarcely necessary to remind you, except for historical purposes, of the

principal change which has been made. I told you that at the close of 1898 we had thirty-one associates; now we have no associates. It was formerly our rule that only the practising analyst, or the analyst having such control of a laboratory as to render him virtually a practising analyst, as distinct from an assistant, was eligible for election to our membership. The consequence was that many of our associates were well qualified chemists approaching middle age, and the position of some of them was becoming every year more and more anomalous; and we had also reason to believe that a number of chemists who could not technically qualify for membership without unduly straining our old rules, had remained outside of the Society rather than join the junior ranks of the associates. In this way, we considered that, if the Society had not actually suffered, it had at all events unnecessarily limited its strength. The range of our work has gradually grown wider, and the very large variety of analytical questions now dealt with in the pages of the ANALYST, and the always increasing circulation of that now indispensable journal, have attracted many friends and sympathizers at home and abroad, to whom we have felt that it would be graceful to open the doors of the Society, should they care to enter into closer and more friendly intercourse with us than existed in merely studying the literature which we placed before them month by month in our journal. We have therefore repealed our old enactment as to the qualifications for membership, substituting a simpler requirement, so that anyone not less than twenty-one years of age, engaged in the profession of analytical chemistry, shall be eligible for proposal for the full membership of the Society, under the conditions laid down in the rules, which will shortly be distributed among you. Additional copies of these you may obtain from the honorary secretaries, in case any of your chemical friends express to you a desire to become acquainted with them.

That the change is not generally distasteful to our old associates may be gathered from the fairly long list of those whose names have been proposed this evening for transference to the rank of members.

I need not detain you by much reference to our financial condition. This has been already ably placed before you by our honorary treasurer. The thoroughness with which he discharges his duties is evidenced on the one side of the account by his success in gathering in our subscriptions, while on the other side, despite the many calls on his purse, he is able to show a sufficient saving to increase, even though not to a very large extent, the Society's funded property. The ANALYST is necessarily an expensive undertaking, and, in addition to our other normal or necessary expenditure, we have during the past year expended a great deal of money in printing and postage, in connection with the distribution among members of Parliament, county councils, borough corporations, and other local authorities, of the views of the Society, as represented by its Council, on matters connected with proposed new legislation on the question of adulteration. The ANALYST, with its present large circulation, is, even from a purely business point of view, no light undertaking; and, with the exception of those who have served on the Council—or rather on the Editorial Committee—few of you probably fully realize the labour necessarily spent, both by our editor, Dr. Sykes, and by our honorary treasurer, Mr. Voelcker, on the mere accounts of the journal,' and matters incidentally relating thereto. The

treasurership of the Society in early days was a very light task, but it has now grown to very serious proportions, and is really a very formidable consumer of the time that is so faithfully and ungrudgingly given to it by the present occupant of what I now regard as the most important office on the Council.

Although I have thus ventured to crave your recognition of the care given to your business affairs by our treasurer and by our editor, it would seem hardly necessary that I should mention, as a self-evident subject for our gratitude, the literary labour spent by the latter on our monthly journal. We have an editorial committee, and that committee works loyally, and works hard, in the duties allotted to it; and we have an industrious and able staff of abstractors. But the success of such a journal must always necessarily depend mainly upon the constant daily work of its editor—work which, in the case of Dr. Sykes, may be almost said to be gratuitous; for, although we give him an honorarium for his services, it is an honorarium which has no relation whatever to the work that he does. Happily, he is a man of leisure, and still more happily, he is willing to devote that leisure to the good cause of editing and managing this journal.

Our editor has had still more to do during the past year than heretofore in the editing of abstracts. I cannot help wishing, however, that we had given him more original papers to edit. During the past year only twenty-one original papers have been read before the Society. Most of these have been published in the ANALYST, together with three other original papers. Of abstracts of papers of interest to the analytical profession, published abroad, or elsewhere than in the ANALYST, we have during the year published 296, as against 283 in 1897, and 246 in 1896.

The following table shows the respective numbers of abstracts in each of these years, published under the various headings of Food and Drugs Analysis, Toxicological Analysis, Organic and Inorganic Analysis, and Apparatus.

ABSTRACTS PUBLISHED IN THE ANALYST.

	1896.	1897.	1898.
Food and Drugs Analysis	55	86	72
Toxicological Analysis	9	5	3
Organic ,, ...	86	87	109
Inorganic ,, ...	86	91	89
Apparatus	9	14	23
	245	283	296

The following were the papers read before the Society during the year:

"Copper 'Pure for Analysis.'" By J. W. Westmoreland.

"Note on the Tests for Distinguishing Boiled from Unboiled Milk." By Henry Leffmann, M.D.

"The Composition of Milk and Milk Products." By H. Droop Richmond.

"The Calculation of Added Water in Adulterated Milk." By H. Droop Richmond.

"Japanese Wood Oil." By John H. B. Jenkins.

"A Typical North-East Lancashire River." By F. R. O'Shaughnessy.

"The Analysis of Marmalade." By L. Kidgell Boseley.

"A New Form of Condenser." By Cecil H. Cribb.

"Water Supply in Relation to the Maidstone Epidemic." By Matthew A. Adams, F.R.C.S.

"Note on Some Apparatus." By H. Droop Richmond.

"Sewage Analysis, and Standards of Purity for Effluents." By C. G. Moor, M.A.

"Note on Certain Resins." By Rowland Williams.

"The Proportion of Oxygen Present in Linseed Oil, both before and after Oxidation." By Rowland Williams.

"Chicory, and Variations in its Composition." By Bernard Dyer, D.Sc.

"The Sulphuric Acid in Portland Cement." By Eug. Ackermann.

"A Curious Meat Preservative." By Alfred C. Chapman.

"A Method for the Quantitative Separation of Acetic and Valeric Acids." By Alfred C. Chapman.

"Contributions to the Chemistry of Drying Oils, with a Method for the Examination of Linseed Oil." By Otto Hehner and C. A. Mitchell, M.A.

"Automatic Pipettes and Burettes." By A. W. Stokes.

"The Effects of Recent Drought on the Quality of Milk." By A. W. Stokes.

"On the Use of the Micro-Spectroscope, and the Methods of Detecting Blood in Chemico-Legal Investigations." (Lecture.) By Alfred H. Allen.

The following original papers were also published in the ANALYST.

"Note on the Volume Concentration of Condensed Milk." By A. McGill.

"Note on the Examination of Liniment of Camphor." By Norman Leonard, B.Sc., and H. Metcalfe Smith.

"The Relation between the Specific Gravity and the Insoluble Fatty Acids of Butter and other Fats." By Norman Leonard, B.Sc.

I ventured last year to point out that many chemists read before other societies, or published elsewhere, papers which I thought ought properly to be read and discussed at the meetings of this Society, which alone of the many chemical societies is the one which is mainly and essentially devoted to the study of chemical analysis. I still think that this is in some sense due to the fact that many suppose that we do not care to discuss here papers dealing with subjects other than those cognate to matters coming before public analysts in connection with their statutory duties under the Sale of Food and Drugs Acts. It has been at various times urged by many members, that we should attract more papers to our Society were we to drop the word "Public" from our title. While much is to be said for, as well as against, such a change in our title, and as the suggestion has been again made quite recently, I would repeat what I think I said at our recent Extraordinary General Meeting, viz., that the proposition to change our name has been long and seriously discussed by your Council, with the result that we have thought it better for the Society to advise you to retain the old colours under which you have now sailed well for five-and-twenty years. I hope, however, that the change recently made in the wording of our rules may induce some who are—not to use the word offensively—guilty of reading elsewhere analytical papers which ought to be read here, to at once seek to become enrolled as members of this Society, and to henceforth read their contributions to the literature of analytical chemistry at our meetings, where they

may surely expect to meet with a fairer appreciation than is often possible in other societies, which deal only occasionally and incidentally with analytical matters.

I ventured last year to express satisfaction that the Institute of Chemistry, under the able presidency of our own late President, Dr. Stevenson, was about to see its way to granting a special diploma in pharmacology and therapeutics, such as would enable candidates for public analystships to produce the evidence of qualification required by the Local Government Board in these subjects as well as in chemistry. The arrangements then in contemplation have been completed, and the Council of the Institute now holds a special examination in these subjects, for such members of the Institute as may care to present themselves. The Institute of Chemistry is now, therefore, able to furnish the full evidence of qualification required by the Local Government Board, without a candidate being obliged to have recourse to any other Society or corporation, whether medical or pharmaceutical, or to merely individual or personal testimony as to therapeutical knowledge, which was always vague and unsatisfactory, and the acceptance of which by the Local Government Board was always a matter of uncertainty and difficulty. Whether the Local Government Board is technically right in demanding therapeutical and pharmacological qualification from the candidate for a public analystship before confirming his appointment is a somewhat vexed question which I do not propose to discuss. The fact exists that the demand is made, and it appears to be satisfactory that we can now meet it from our own recognised chemical diploma-granting body, viz., the Institute of Chemistry. Certainly no public analyst can be the worse for a fair knowledge of crude drugs, even though their examination may not come within the scope of his normal duties; and it certainly appears to be educationally healthy and desirable that a student who is taking, as his final examination for the associateship of the Institute, the section which relates more especially to the analysis of food and drugs, should learn sufficient practical materia medica to enable him to pass this examination.

Not the least interesting to analysts among the chemical events of the year has been the completion, by the issue of its last volume, of the great work of our esteemed Past President, Mr. Allen, "Commercial Organic Analysis," a work which, whatever modification or extension it may have to undergo in the future, as our science develops, must necessarily live as one of the classical achievements of chemical literature, preserving the name of Allen side by side with the names of Gmelin, Watts, and Fresenius. The undertaking has been almost a stupendous one, having regard to the rapid daily growth of the subjects of which it treats. In 1875 the most comprehensive treatise of the kind, then fairly up to date, was Prescott's little manual on "Proximate Organic Analysis," a book of only about 180 small octavo pages. A glance through that little book, to the eye now familiarized to the use of Allen's splendid manual, perhaps brings home to one more cogently and more concentratedly than anything else the vast progress made by analytical chemistry during the last five-and-twenty years. Allen has not been merely the chronicler of this progress. He has largely been a contributor to it, not merely by the contributions emanating from his own laboratory, which have been many and valuable, but by the educational and stimulating influence of the publication of his book. I like

to think that, if not actually responsible for the birth of Allen's book, the Society of Public Analysts has nevertheless had no small share in indirectly encouraging and influencing its progress and development. Although, as it appeared volume by volume, it has appealed to a world-wide circle of readers, yet I venture to think that the appreciation and personal encouragement which he has found in the little brotherhood of chemical friends banded together in this Society, have done not a little towards enabling its author to persevere unflinchingly in the heavy task to which he has devoted the best years of his life; and I like to think, when reviewing the great progress made in proximate organic analysis during the last quarter of a century, that, in many directions, the lines of investigation, abroad as well as at home, were first marked out by the early work of members of this Society.

The question which has engaged the attention of the Society now for some years still remains unsettled—I mean, the question of reformed legislation with regard to food adulteration. A year ago the only progress to be chronicled was that a Bill had been introduced into Parliament by the President of the Local Government Board, at the very end of the session of 1897, and simultaneously withdrawn. The Bill was a fragmentary and incomplete one, which failed to deal with the most important of the recommendations contained in the report of the Select Committee of the House of Commons on Food Products Adulteration, and was admittedly only put forward as a tentative measure. Your Council deemed it wise to criticise this Bill somewhat severely; and the Bill met with so much severe criticism from so many other directions that it was not reintroduced. The President of the Local Government Board appears to have then come to the conclusion that the subject was too knotty a one to be dealt with as a whole, and he accordingly, after another session's reflection, proposed—and, as before, simultaneously withdrew—another tentative measure, based on quite different lines, in which he proposed to deal only with agricultural products, to the exclusion of food articles of general manufacture. Such a Bill necessarily left undealt with a great proportion of the recommendations of the Food Products Adulteration Committee, though it partially embodied the spirit of the recommendation with regard to a standing authority to lay down standards or limits of composition. The Bill contained the following clause:

> The Board of Agriculture may, after such inquiry as they deem necessary, make regulations for determining the extent to which any deficiency in any of the normal constituents of milk or butter, or the presence of any foreign matter in any sample of milk or butter, shall raise a presumption, until the contrary is proved, that the milk or butter is adulterated or impoverished.

I take it that the intention was that the Board of Agriculture should from time to time, as it deemed advisable or necessary, itself appoint a committee to take evidence and make recommendations; and it is quite possible, if the only articles to be dealt with were milk and butter, that the Board of Agriculture, acting with the assistance of a departmental committee appointed by itself, might satisfactorily take the place of the comprehensive standing committee of reference recommended in the report of the Select Committee. But, in the Bill of 1898, no machinery was provided for discharging the many other functions, connected with food generally,

which it was proposed should be assigned to the committee of reference contemplated in the Select Committee's report. It has been recently announced, on Government authority, that the President of the Local Government Board has handed over the charge of the Bill to the President of the Board of Agriculture. Mr. Walter Long, in a recent speech, announced his intention of introducing during the coming session a Bill which he hoped would reduce adulteration, though from his speech it is to be feared that it will again be a Bill dealing only with food products produced by farmers. Mr. Long, however, stated, in effect, that he had been much struck by the general unanimity of the demand for fresh legislation, and that he had been approached by traders of all kinds, and by various commercial associations, far more than by agricultural interests. He was assured that there was wholesale adulteration going on in the country, and believed there was truth in the statement. He further said that it was a remarkable fact that all the statements which were made were on one side, and that no one had suggested that there was another side to the question, every assertion being to the effect that there was a great deal of fraud being perpetrated. He was satisfied that it was not a state of things that ought to be allowed to continue, and it was the intention of the Government to introduce, through himself, in the coming session, a Bill which he hoped would enable them to deal satisfactorily with the question. Perhaps it is not too late to hope that Mr. Long will yet find himself able to enlarge Mr. Chaplin's Bill in such a way as to deal comprehensively with the question. Such a Bill we all believe must ultimately come, and to deal with the whole question piecemeal is only to prolong the unsettled condition of affairs which has been so long causing concern to so many sections of the public. The mere question of the regulation of the use of dangerous preservatives and colouring matters is at the present time so vexed a one and so pressing a one that it would be anomalous if any Bill were introduced which did not hold out some satisfactory means of dealing with it.*

The Council of this Society has more than once had its attention directed to the action of certain County Councils in undertaking to make, for the general public, in their own laboratories and at the hands of their own exclusively-engaged public analysts or medical officers, at the expense of the county rates, analyses of water and other articles at merely nominal charges, thus entering into competition with the ordinary practitioner of analytical chemistry. The Council has obtained from the Local Government Board a distinct expression of opinion to the effect that County Councils, in applying the county rates for such purposes, are exceeding their statutory powers. It is sincerely to be hoped that the effect of this declaration will be to put an end to so unfair and so unwarrantable a form of competition.

I cannot close this somewhat random dissertation without referring to the very pleasant summer meeting, in which so many of us joined, at Woburn, where Dr. J. A. Voelcker was good enough to show us over the experimental farm which has been under the charge successively of his late father and of himself for over twenty years. In historical interest the Woburn farm has now become second only to the older station at Rothamsted, and many of our members were pleased to

* Since this address was delivered Mr. Long has introduced his Bill (February 23) into the House of Commons. The scope of the Bill is wider than that of the Agricultural Bill introduced last year.

have the opportunity of visiting it. The recently established pot-culture station, established for the purpose of carrying out investigations on the influences of various chemical substances on plant growth, under the bequest of the late Mr. Hills, was very interesting to most of us; and the kindness of the Duke of Bedford in throwing open to our inspection the art treasures of Woburn Abbey added pleasantly to the attractions of a most enjoyable meeting.

Now a brief personal word. I close this evening an official connection with the Society extending over sixteen years, fourteen years as one of your secretaries, and two years as your President. And I feel that it would be unbecoming did I not now, in taking my official farewell of you, say how much of the happiness of my life has been due to friendships formed in this Society, and did I not also make some formal acknowledgment of the much kindness—and often, I fear, much forbearance—which you have extended to me in the face of many shortcomings, some of them conscious, and some of them (and usually these are the worst) not conscious. Not to make blunders, however, is a gift proverbially reserved for those who make nothing. Mine you have been good enough year after year to overlook, and my best consolation is that the Society has flourished in spite of them. My seat in this chair I now resign to an able and accomplished successor. My old secretarial chair, which during the last two years has been occupied by Mr. Cassal, will from this evening be occupied by a gentleman who is not a very old member of the Society, but who is a very loyal one. Mr. Chapman is a chemist of distinguished ability and a man of energy and industry, and he will have a good trainer in his new duties in my old colleague, Mr. Bevan, who still happily retains his post.

ANNUAL DINNER OF THE SOCIETY OF PUBLIC ANALYSTS.

The annual dinner was held at the Criterion Restaurant, after the annual meeting, under the chairmanship of the newly-elected President, Mr. W. W. Fisher, M.A. About sixty members and guests were present. Amongst the latter were Professor Odling, F.R.S.; Professor Tilden, F.R.S.; Dr. Thos. Stevenson, President of the Institute of Chemistry; Mr. Walter Hills, President of the Pharmaceutical Society; Mr. George Beilby, President of the Society of Chemical Industry; Dr. Childs; Dr. Longstaffe; Dr. Pakes; Mr. E. Holmes; Mr. F. W. Beck; and Mr. Aubrey W. Rake.

The following toasts were proposed: "The Queen," by the President; "The Institute of Chemistry," by Professor Odling, F.R.S., responded to by Dr. Thos. Stevenson, President of the Institute; "Kindred Societies," by Dr. Bernard Dyer, responded to by Mr. Walter Hills, President of the Pharmaceutical Society; "The Society of Public Analysts," by Professor Tilden, F.R.S., responded to by the President; and "The Guests," by Mr. Otto Hehner, responded to by Mr. George Beilby, President of the Society of Chemical Industry.

SECTION OF BORE-HOLE AT ILKESTON.

The lettering in these diagrams can be readily deciphered by the aid of a weak magnifying-glass.

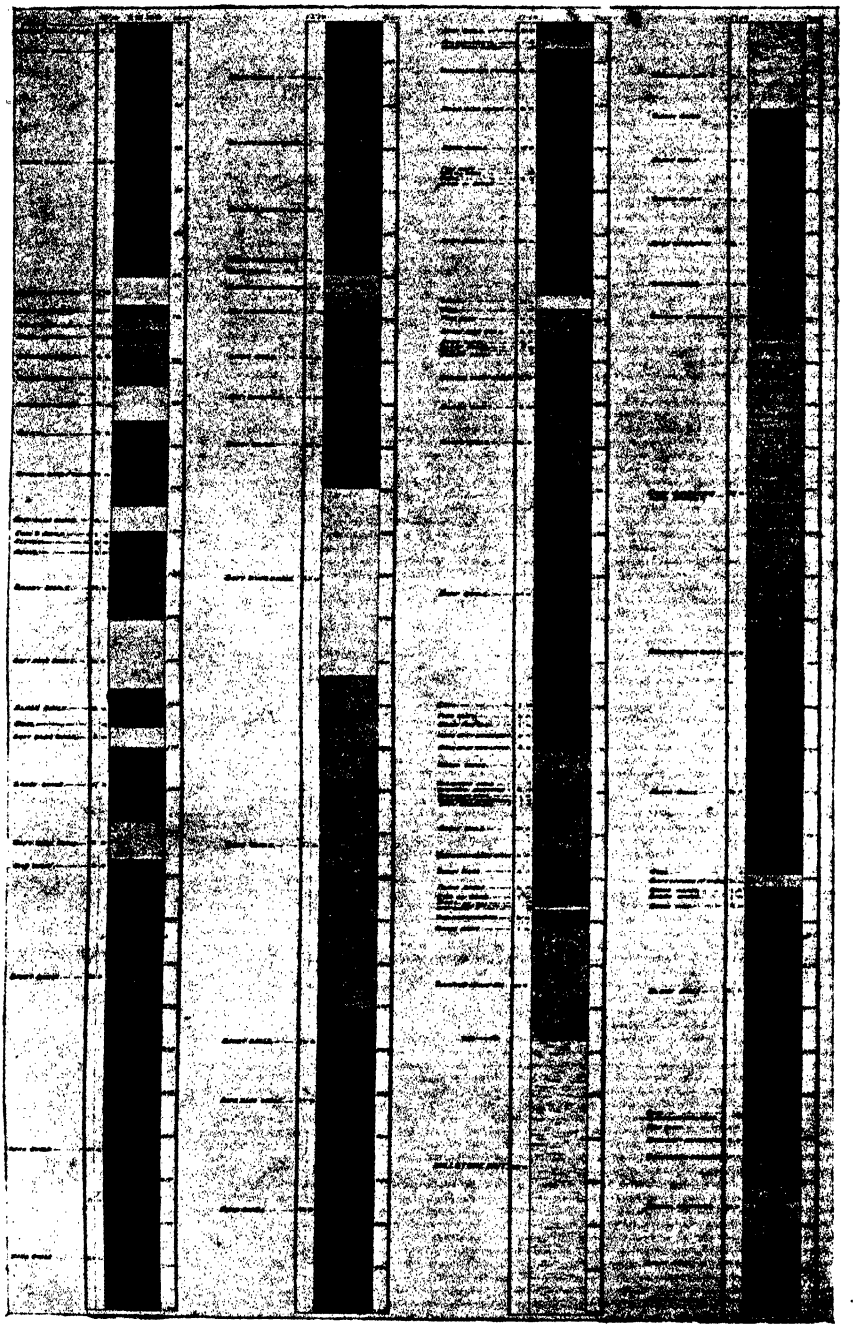


DIAGRAM No. 1.

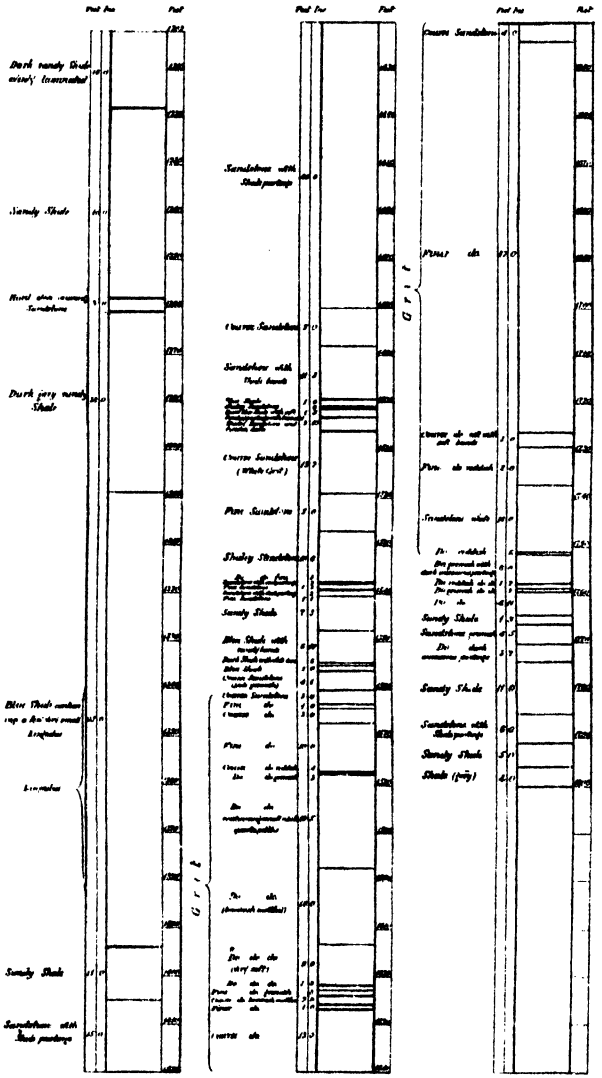


DIAGRAM No. 2.

THE OCCURRENCE OF BARIUM COMPOUNDS IN ARTESIAN WELL WATER.

By John White, F.I.C.

(*Read at the Meeting, January* 4, 1899.)

The presence of a salt of the metal barium in solution in a natural water in this country is in my experience of very rare and unusual occurrence. I can only find a few instances recorded of the existence of a soluble salt of this metal as a constituent of unartificial waters, and therefore think it desirable to lay before this Society the results of my analyses of some samples of water derived from an artesian well of very great depth at Ilkeston in Derbyshire. I give all the information I have been able to obtain respecting the bore-hole, and exhibit drawings in section of the strata passed through, which are here reproduced (see diagrams 1 and 2). For much of this information I am indebted to Mr. Henry J. Kilford, the Borough Surveyor of Ilkeston, also for the loan of the drawings.

The bore-hole, the surface of which is 160 feet above sea-level, O.D., has been carried to a total depth of 1,801 feet, water being found at 837 feet, and rising to a height of 15 feet above the surface. As only about 180 gallons per hour were obtained at the surface from that depth, the bore was deepened as stated, with the result that the yield remained practically unaltered. A pumping shaft was then sunk round the bore-hole to a depth of about 150 feet, and this is now in course of being deepened to about 300 feet. The steel tubes were removed as the sinking proceeded, and a considerable increase in the volume of water yielded has been the result, great difficulty being experienced at times in plugging up the tube. It was during the removal of a length of tube that the accident happened which caused the admittance to the bore-hole of the abnormal water presently to be described. Mr. Kilford is of opinion that the tubes split somewhere lower down, and by sinking the shaft below the Kilburn coal measures, he expects to be able to "tub out" the abnormal water. The bore-hole was lined with steel tubes to a depth of 1,040 feet.

The diagrams give exact particulars of the nature and thickness of each layer of the strata passed through. By adding together the figures given for the various layers of like material, and grouping them according to their general description and classification, arough synopsis of the formation is obtained, as shown in the following table:

Table I.

Nature of Strata.	Varieties mentioned in Section Diagram.	Total Thickness in feet.
Shale	Black, blue, light blue, dark blue, soft blue, gray, dark, sandy, variegated.	1,100
Sandstone ...	Brownish mottled, broken, coarse, close-grained, fine, greenish gray, dark gray, hard gray, open gray, reddish, shaley, streaked, variegated, white.	350
Millstone grit ...	— — — —	300
Coal	— — — —	17
Fire clay	— — — —	16
Miscellaneous ...	Beryl, blue bind, sandy bind, clay, clunch, ironstone, limestone, spar.	20

Before proceeding further to consider the nature of the strata, I give in the next table the results of my analyses of six samples of water :

A is the water originally yielded by the bore-hole.

B is the abnormal water which gained access to the bore-hole, as is supposed by the splitting of the steel lining tubes.

C is a similar sample, collected about six months later than B.

D is from the pumping shaft, collected since this abnormal water appeared.

E and F are from the pumping shaft, taken about 80 feet below the surface, before the abnormal water was found.

TABLE II.

Results expressed in Parts per 100,000.

	From Bore-hole.			From Pumping Shaft.		
	A.	B.	C.	D.	E.	F.
Total solid matter, dried at 100° C.	59·5	2628·0	2752·0	282·0	62·5	51·0
Loss on ignition	3·0	194·0	198·0	19·0	—	—
Saline residue	56·5	2434·0	2554·0	263·0	—	—
Free and saline ammonia... ...	0·060	0·875	—	0·120	0·100	0·080
Albuminoid ammonia	0·002	0·004	—	0·006	0·008	0·004
Nitrogen in nitrates and nitrites	0·0	0·0	0·0	0·0	0·055	0·055
Oxygen absorbed in four hours at 80° F.	0·036	0·680	—	—	0·124	0·035
Hardness by soap test :						
Temporary	—	—	—	21·03	13·58	13·26
Permanent	—	—	—	23·22	7·02	7·14
Total	7·71	—	—	44·25	20·60	20·40
Silica	0·0	0·0	1·2	1·0	—	—
Iron oxide, Fe_2O_3	Traces.	4·5	1·6	1·2	Traces.	Traces.
Barium oxide, BaO	1·38	28·36	29·94	2·23	0·0	0·0
Calcium oxide, CaO	2·60	141·64	169·0	15·40	—	—
Magnesium oxide, MgO	Traces.	42·16	39·06	4·82	—	—
Potassium oxide, K_2O	0·0	0·0	0·0	0·0	—	—
Sodium oxide, Na_2O	—	1080·76	1109·15	114·02	—	—
Heavy metals	Absent.	Absent.	Absent.	Absent.	Absent.	Absent.
Chlorine	17·1	1491·0	1554·9	142·0	10·7	4·8
Sulphuric anhydride, SO_3 ...	0·0	0·0	0·0	0·0	—	—
Phosphoric anhydride, P_2O_5 ...	0·0	0·0	0·0	0·0	—	—
Total alkalinity, calculated as $CaCO_3$...	26·5	20·0	19·0	25·0	34·5	34·0
Alkalinity after boiling, calculated as $CaCO_3$	21·25	2·0	1·5	2·5	21·5	21·0
Alkalinity after boiling, equivalent to Na_2CO_3	22·52	—	—	—	22·79	22·26
Specific gravity at 60° F. water 1·000	—	1·018	1·019	—	—	—
Appearance in 2-feet tube ...	Fairly clear ; brownish-green.	Turbid ; brownish-green.	Turbid ; brownish-green.	Turbid and opalescent ; brownish-green.	Rather turbid and opalescent ; brownish-green.	Rather turbid and opalescent ; brownish-green.
Odour	None.	Slight.	Slight.	None.	None.	None.
Taste	None.	Strongly saline.	Strongly saline.	Saline.	None.	None.

These waters are of peculiar and abnormal composition. Samples E and F are of no great interest, and are only included as indicating the kind of water occurring in the neighbourhood of the bore-hole; they are typical examples of water such as is frequently found in or near the coal measures, containing, as these samples do, a comparatively high amount of free ammonia and chlorine, with traces of iron and a considerable quantity of sodium carbonate.

The first four samples contain barium in varying amount, and there is consequently an entire absence of sulphates and phosphates. As compared with the total solid matter present, the proportion of chlorine is relatively exceedingly high, while the lime and magnesia are in each instance very low. There is no evidence of pollution, the free ammonia being doubtless of mineral origin, and organically the waters may be pronounced to be pure.

Sample A was decidedly alkaline from presence of sodium carbonate, a fact which renders the occurrence in the water of barium particularly remarkable, inasmuch as sodium carbonate will precipitate the chloride or other soluble salt of barium at the ordinary temperature and in very dilute solutions. The barium, therefore, must exist as the carbonate, which salt is slightly soluble in water; it is present to the extent of 1·77 parts per 100,000.

This water flowed from the bore-hole at a temperature above normal; at the date of collection the thermometer in the open registered 47° F., but the temperature of the water was 61° F.

Samples B, C, and D all contained barium chloride. B and C closely resemble each other in their phenomenal composition; they contain an amount of barium corresponding respectively to 38·55 and 40·70 parts per 100,000 of barium chloride. With the exception of a water stated by Professor Bedson (*J.S.C.I.*, vol. vi., p. 712) to contain 96·04 grains per gallon of barium chloride, this is the highest amount I have been able to find recorded as occurring in a natural water. The other features of these waters worthy of notice are the extraordinary quantity of common salt in solution, and the comparatively small amounts of lime and magnesia present.

When freshly obtained, samples B and C, upon being briskly shaken at the ordinary temperature, evolved a considerable amount of gas, which I believe mainly consisted of nitrogen.

Sample D appears to consist of some similar water to B or C, diluted to nearly ten times its volume with some other normal water; the solid matter, chlorine, barium chloride, lime and magnesia, are present approximately in about one-tenth the amount in which these constituents occur in samples B and C.

In Table III. I have combined the acids and bases to show what may probably represent the actual salts present in solution in the four waters containing barium. It frequently happens that there is a difference of opinion as to the method to be adopted in combining these acids and bases I have no wish, therefore, to insist upon the correctness of the view I have taken, but simply to follow the usual custom and state the calculated analyses:

TABLE III.

Results expressed in Parts per 100,000.

Calculated Analysis.	A.	B.	C.	D.
Barium carbonate	1·77			
Calcium carbonate ...	4·64			
Sodium carbonate	22·52			
Sodium chloride	28·17			
	57·10			
Silica	—	0·0	1·20	1·00
Oxide of iron	—	4·50	1·60	1·20
Barium chloride	—	38·55	40·70	3·03
Calcium chloride	—	258·55	315·08	2·77
Calcium carbonate ...	—	20·00	19·00	25·00
Magnesium chloride ...	—	100·13	92·76	11·44
Sodium chloride	—	2039·50	2093·08	215·29
	—	2461·23	2563·42	259·73

The barium was estimated in the water without previous concentration by acidifying with dilute hydrochloric acid, and then precipitating with very dilute sulphuric acid (about 1 in 300) and heating to boiling. The filtrate and washings were subsequently used for the determination of the lime by precipitation with ammonium oxalate in the usual way.

Having proved the absence of all other metals, including potassium, the amount of sodium oxide was ascertained, which was equivalent to the sodium chloride calculated. This was then checked by sulphating the residue from a known quantity of water, igniting and weighing, and comparing the result with the sum of the salts calculated to their respective sulphates. In this way a very fair approximation was obtained.

These waters give, as I will show you, marked precipitates with potassium chromate and dilute sulphuric acid, without previous concentration. I would point out in this connection that it is not advisable, when testing waters for lead, to rely entirely upon the chromate or bichromate of potash test. This should obviously be confirmed by the use of sulphuretted hydrogen, otherwise such waters as these might be pronounced to contain lead.

And now a few words as to the possible origin of the barium salts. There is not very much of a striking character, from a geological point of view, to be discovered by an examination of the diagram of the section of the strata through which the bore-hole passes, and no direct evidence appears to be afforded as to the whereabouts of the barium compounds. Some of the seams are described as containing "spar," but I have been unable to obtain any of this for analysis, and cannot say what is its composition. Nearly one-fifth of the strata is classified under the head of sandstone, and, with millstone grit, this constitutes upwards of one-third of the whole. Clowes (*Chem. News*, 52, p. 194) states that the beds of the new red sandstone near Notting-

ham are permeated by minute crystals of barium sulphate, which acts as a cementing material. Dieulafait is also stated to have shown that all primary rocks contain barium in sufficient quantity to be easily detected. The occurrence, however, of barium sulphate in the strata, although the most likely salt to be present, does not of itself account for the barium carbonate or chloride in these waters. The only explanation that occurs to me is that the barium sulphate in the rock, etc., has by some means, possibly by the action of heat in presence of carbon or carbonaceous material, been reduced to the sulphide, and this salt has then, by coming into close contact with beds of common salt or with some strong solution of brine, been converted into the chloride. This might then react with other water containing sodium carbonate, and so become changed into barium carbonate. It is possible that the carbonate might be directly produced from the sulphide, but the first hypothesis, having regard to the enormous amount of common salt associated with the barium salts, seems to me to be the more probable.

Clowes (*Royal Society Proceedings*, vol. xlvi.) gives the results of some analyses of deposits formed in the water-boxes of coal-mines in the neighbourhood of Newcastle-upon-Tyne. These deposits consisted mainly, and in some cases almost entirely, of barium sulphate; they were soft and easily powdered, and appeared to have been produced by a process of rapid precipitation. He is of opinion that these deposits may be formed by the admixture of a water containing barium chloride, such as the one to which I have already referred, communicated by Bedson, with a water containing sulphuric acid or ferrous sulphate, produced by the oxidation of pyrites in the coal measures. The water analysed by Bedson was from a coal-mine near Newcastle-upon-Tyne.

Neither Clowes nor Bedson, as far as I know, have, however, offered any explanation of the origin of the barium chloride in solution in these waters.

Another instance of the occurrence of barium chloride in a natural water is to be found in a spring at Llangammarch, a small village in Breconshire, Central Wales. This spring has been analysed by Dupré and by S. Arch Vasey, of the *Lancet* laboratory. The former found 6·26 and the latter 6·74 and 6·49 grains per gallon of barium chloride, the total mineral matter amounting to 302 grains per gallon. Waters have also been found containing this salt at collieries near Sunderland and near Rotherham, and in a spring water near Shotley Bridge in Durham.

Barium chloride is said to possess valuable medicinal properties, acting chiefly upon the heart. As it is not, however, an official drug of the British Pharmacopœia, it is possible that its therapeutical effects are not yet properly understood. According to toxicological authorities, the minimum authenticated fatal doses recorded are of the carbonate 60 grains, and of the chloride 100 grains. From the results I have laid before you, it does not seem probable that cases of poisoning by the consumption of water containing salts of barium are likely to arise.

Discussion.

The President thought that it might be possible to account for the presence of the soluble barium somewhat less elaborately than in the manner suggested by Mr. White, viz., simply by the action of sodium carbonate, under pressure, on the

barium sulphate. Without pressure, of course, the action of aqueous solutions of alkaline carbonates on barium sulphate was very slight, even when the solutions were boiled with the sulphate. It might be, however, that the original material was barium carbonate (witherite), which would more easily be dissolved. In the red sandstone, strontium occurred as celestine, the sulphate, and as strontianite, the carbonate; and possibly barium also occurred there in the forms of sulphate and carbonate. Anyone who met with either of these waters would at once notice that there was something unusual about it, from the fact that it contained no sulphuric acid.

Mr. HEHNER inquired whether the author had examined the water spectroscopically for the presence of small quantities of strontium as well as barium. In the case of many analyses made by Fresenius it had been demonstrated that both of these metals were often present in water, of course in very small quantities, and not in any way comparable with the cases referred to in the paper.

Dr. RIDEAL remarked that an analysis made by Dr. Dupré of the Breconshire water referred to had been widely circulated for purposes of advertising the water as a specific for cardiac diseases; and, inasmuch as that water only contained six or seven grains of barium per gallon, it would appear that these Ilkeston waters, containing several times that quantity, should have a greater value in such diseases. It would be noted that the absence of alumina was a characteristic feature of all these waters.

Mr. WHITE, in reply, said that the President's suggested explanation did not appear to account for the presence of barium chloride, and he (Mr. White) thought the barium could not exist in the water otherwise than as chloride. It might, however, account for the carbonate present in sample A, which was the original water from the borehole. The only evidence he had upon which to base his conclusion that barium sulphate was the original mineral, was the information contained in the papers of Dr. Clowes. Dr. Clowes had taken much interest in the subject, and it seemed quite clear that, in the neighbourhood referred to, there existed considerable quantities of barium sulphate in the sandstone, which formed about a fifth of the total strata passed through in boring the well; and therefore he (Mr. White) thought it fairer to conclude that barium sulphate, rather than barium carbonate, was present originally, for there was no evidence at all of the occurrence of barium carbonate in the neighbourhood. Some few miles away there was a piece of millstone grit, forced up, probably, by volcanic action, known as the "Hemlock Stone," from its shape and spreading top. Dr. Clowes had published an analysis of this stone, which showed that it contained in some parts a large percentage of barium sulphate. He (Mr. White) could confirm this from his own analysis of portions of the stone. He had not used the spectroscope to prove the absence of strontium, but had tested every precipitate of barium sulphate very carefully, without getting any evidence of the presence of strontium. He had no information as to the solubility of barium carbonate in solutions of alkaline chlorides, but had already, in referring to the President's remarks, stated his reasons for assuming the original presence of barium sulphate, and the way in which he thought the chloride was to be accounted for.

ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

FOODS AND DRUGS ANALYSIS.

Testing for a Yellow Azo-colour in Fats, etc. A. H. Low. (*Journ. Amer. Chem. Soc.*, vol. xx. [11], 889.)—A few c.c. of the pure filtered fat are treated with an equal volume of a mixture of strong sulphuric acid, 1 part, and glacial acetic acid 4 parts, the whole being then heated nearly to boiling, and thoroughly mixed by agitation. After settling, the lower stratum (mixed acids) will exhibit a strong, wine-red coloration, if the yellow azo-dye be present, whereas only a very faint brownish tinge is produced in the case of pure butter. The sulphuric acid may be replaced by strong hydrochloric acid, or a mixture of 1 part strong sulphuric acid with 3 parts water may be employed; the acetic acid mixture, however, gives the best results.

C. S.

The Detection of Horseflesh in Sausages. T. Bastien. (*Journ. Pharm. Chim.*, 1898, vii., 540-542.)—The author has examined various methods based on the colour reaction which the glycogen of the horseflesh gives with iodine. With the original process of Bräutigam and Edelmann (ANALYST, xix., 24) he has only been able to obtain certain results in the presence of a large proportion of horseflesh, and he has also found Courlay and Coremon's modification (ANALYST, xxi., 231) inconclusive.

By varying the experimental conditions he has worked out the following modification, by which, he asserts, it is possible to detect the presence of 5 per cent. of horseflesh in sausages, even in the presence of starch: About 20 grammes of the finely minced sausage are boiled for from thirty minutes to one hour with 100 c.c. of water, so that the volume of the liquid is reduced to about 30 c.c. When cold the broth is filtered, and about 10 c.c. tested with 2 or 3 drops of a solution of iodine in potassium iodide (iodine, 1 gramme; potassium iodide, 2 grammes; water, 100 c.c.) or of iodine water. A fugitive reddish violet coloration indicates the presence of horseflesh. The iodine reagent must be added carefully, since a slight excess changes the colour to reddish-brown.

If starch is also present, the broth is decanted when cold, and from 1 to 2 volumes of acetic acid added according to the quantity of starch. After standing for 5 minutes the liquid is filtered, and 10 c.c. of the filtrate tested as before with 2 to 3 drops of the iodine solution, which in this case also produces a violet colour in the presence of horseflesh.

A summary of the author's experiments is given in the subjoined table:

Pork.	Horse-flesh.	Starch.	Volume of Water.	Duration of Boiling.	Iodine Water.	Iodine in KI Solution.	Addition of 1 vol. of Acetic Acid.		Addition of 2 vols. of Acetic Acid.	
							Iodine Water.	Iodine in KI.	Iodine Water.	Iodine in KI.
Grammes.	Grammes.	Grammes.	c.c.	Hours.						
0	0	0·5 F.	150	½	Dark blue.		Slight brown.	Nil.	Nil.	Nil.
0	0	1·0 F.	150	½	Do.		Do.	Do.	Slight brown.	Do.
0	40	0	150	½	Dark reddish violet.		Blood red.	Blood red.		
0	40	1·0 B.	150	½	Dark blue.		Reddish violet.	Reddish violet.	Reddish brown.	Reddish brown.
0	40	1·0 F.	150	½	Do.		Do.	Do.	Do.	Do.
0	20	0·5 B.	150	¼	Do.		Brownish violet.	Do.	Violet.	Brownish yellow.
0	20	1·0 B.	150	Liquid reduced to half its volume.	Do.		Do.	Reddish brown.	Violet brown.	Do.
0	20	1·0 F.	150		Do.		Do.	Do.	Do.	Do.
0	20	0·5 F.	100	¼	Do.		Reddish violet.	Reddish violet.	Brownish yellow.	Nil.
0	20	0·5 F.	100	½	Do.		Do.	Do.	Do.	Do.
20	0	0	150	½	Nil.					
19	1	0	150	½	Brownish violet.		Yellowish brown.	Nil.		
18	2	0	150	½	Dark reddish violet.		Reddish violet.	Yellowish brown.	Reddish brown.	Reddish brown.
18·5	1	0·5 F.	150	½	Dark blue.		Violet.	Reddish violet.	Light brown.	Faint brown.
18	1	1·0 F.	150	½	Do.		Do.	Do.	Dark brown.	Dark brown.
17·5	2	0·5 F.	150	½	Do.		Do.	Do.	Brownish.	Do.
17	2	1·0 F.	150	½	Do.		Do.	Do.	Do.	Do.

B = wheat starch. F = Potato starch. C. A. M.

ORGANIC ANALYSIS.

Emetine Octo-iodide and the Extraction and Estimation of Alkaloids generally. H. M. Gordin and A. B. Prescott. (*Amer. Journ. Pharm.*, 1899, lxxi., 14-21.)—The authors consider that the simplest and quickest method of obtaining the alkaloidal solution sufficiently free from foreign matter for titration by their periodide method (ANALYST, xxiii., 324) is that of A. B. Lyons (*Manual of Pharm. Assaying*, p. 20). This consists in macerating a weighed quantity of the powdered drug in a definite volume of Prollius' fluid with frequent shaking for several hours, drawing off an aliquot portion of the clear liquid, evaporating and taking up the residue with acidulated water. To obviate the loss by evaporation of the volatile solvent, which often causes too high results, the authors prefer as a general method of extraction a process which in its main features is the same as that used by them in the assay of opium (ANALYST, this vol., 8).

This method has given very good results with all the drugs examined, with the exception of ipecacuanha root. It was found to be almost impossible to extract free emetine completely from this root by percolation with the ethereo-ammoniacal mixture.

Ether, chloroform, and acetone were also tried as solvents, but all yielded results much lower than those obtained by Lyons' process.

Emetine appears to form two periodides, that produced when the iodine is added to the alkaloid differing from that formed when the alkaloid is added to the iodine. The higher periodide, which is the only one the authors have yet examined, is obtained in the latter way. It is a dark brown powder, hardly soluble in benzene, ether, or chloroform, readily soluble in alcohol, and very soluble in a mixture of 4 parts of alcohol and 1 part of chloroform, although chloroform by itself hardly dissolves it. From the results of their analysis they conclude that it is emetine hydriodide hepta-iodide, $C_{28}H_{40}N_2O_5.HI.I_7$, and from this the iodine factor for emetine is calculated to be 0·5453.

The following table gives the comparative results of the gravimetrical and iodometrical assay of various drugs. Except in the case of the ipecacuanha root, for which Lyons' method was used, the alkaloids were extracted by the authors' general method, and titrated as described in their former paper (ANALYST, xxiii., 324). For nux vomica the mean factor of strychnine and brucine (0·47845) was taken, and for atropine the factor 0·2849.

Drug.	Method.	Grammes taken.	Iodine Consumed. Gramme.	Percentage of Alkaloids. Iodometric.	Percentage of Alkaloids. Gravimetric.
Nux vomica	Iodometric 1	1	0·0526816	2·52	
	,, 2	1	0·0526725	2·52	
	Gravimetric 1	1	Alkaloids shaken out and weighed.	—	2·73
	,, 2	1		—	2·73
Belladonna root	Iodometric 1	2·5	0·0459179	0·52	
	,, 2	2·5	0·0459263	0·52	
	Gravimetric 1	2·5	Alkaloids shaken out and weighed.	—	0·51
	,, 2	2·5		—	0·51
Belladonna leaves	Iodometric 1	5	0·0478286	0·27	
	,, 2	5	0·0475922	0·27	
	Gravimetric 1	5	Alkaloids shaken out and weighed.	—	0·28
	,, 2	5		—	0·28
Ipecacuanha root	Iodometric 1	2	0·0957764	2·61	
	,, 2	2	0·0986635	2·69	
	Gravimetric 1	2	Alkaloids shaken out and weighed.	—	2·63
	,, 2	2		—	2·62

C. A. M.

The Separate Estimation of Strychnine and Brucine. H. M. Gordin and A. B. Prescott. (*Amer. Journ. Pharm.*, 1899, lxxi., 18, 19.)—The periodide method of assaying nux vomica (ANALYST, xxiii., 324) may be used in conjunction with Dunstan and Short's method of separating strychnine from brucine (*Pharm. J. Trans.* (3), 14, 290) as a convenient method of separately estimating these alkaloids in the drug. The acidulated alkaloidal solution obtained from 4 grammes of nux vomica in

any suitable way is made up to 100 c.c. Fifty c.c. of this are run into an Erlenmeyer flask, 10 c.c. of 2 per cent. sulphuric acid added, and water to about 200 c.c. After the addition of 25 c.c. of a 5 per cent. solution of potassium ferrocyanide, the flask is closed and shaken continuously for about thirty minutes. The liquid is filtered and the precipitate washed with water containing 1 per cent. of sulphuric acid, until the washings diluted with water have no bitter taste.

The filter is then pierced, the precipitate washed into a second flask, where it is mixed with 20 c.c. of a 5 per cent. solution of zinc sulphate, and the flask kept on a boiling water-bath for about fifteen minutes. The zinc sulphate decomposes the strychnine ferrocyanide, zinc ferrocyanide being precipitated and strychnine sulphate left in solution. When completely cold the liquid is diluted to 100 c.c. Of this 50 c.c., representing 1 gramme of nux vomica deprived of the brucine, are filtered off and run into a flask containing 20 c.c. of decinormal iodine solution and about 2 c.c. of dilute hydrochloric acid. The amount of iodine consumed multiplied by 43·9 (100 times the strychnine factor) gives the percentage of strychnine.

The iodine consumed by the total alkaloids is determined in another portion of the alkaloidal extract in the same way, and the difference between the two amounts multiplied by 51·79 (100 times the brucine factor) gives the percentage of brucine in the drug.

The following results were obtained by this method with a test-solution containing 0·16 per cent. of strychnine and 0·22 per cent. of brucine (anhydrous):

Iodine consumed by 10 c.c., before the removal of Brucine. Gramme.	Iodine consumed by 10 c.c., after the removal of Brucine. Gramme.	Found, per cent.	
		Strychnine.	Brucine.
0·0843130	0·032397	0·14	0·24
0·0843132	0·032397	0·14	0·24

C. A. M.

Melzer's Picrotoxin Colour-Reaction as a Test for Cholesterin and Phytosterin. H. Kreis. (*Chem. Zeit.*, 1899, xxiii., 21.)—Melzer has shown (*Zeits. anal. Chem.*, 1898, xxxvii., 351 and 747) that when picrotoxin is treated with benzaldehyde and sulphuric acid a red colour is produced; the present author finds that cholesterin and phytosterin (indifferently) yield an analogous reaction. A few drops of a dilute ethereal solution are allowed to evaporate in a porcelain basin, when two drops of Melzer's alcoholic solution of benzaldehyde and one drop of strong sulphuric acid are added. The mixture is made to spread itself over the surface of the basin and then left to rest. Cholesterin and phytosterin develop a reddish-violet colour, afterwards changing to a dark violet; picrotoxin gives rather an "eosin-red" tint. When the colour disappears, it may be restored by more acid. The original tints shown by the fats resemble that produced in the Hager-Salkowsky test for cholesterin, for the colouring-matter is also soluble in chloroform, and may be the same substance. The latter reaction succeeds equally when Melzer's benzaldehyde is replaced by chloroform.

In view of this similarity, the author remarks that caution must be observed in toxicological analysis where picrotoxin has been apparently identified by its colour-

reactions; in the ethereal extract of the acid liquor obtained after destruction of normal organic matter by the Stas-Otto method, cholesterin may well be present.

F. H. L.

On some Recent Methods for Examining Balsams and Resins. K. Dieterich. (*Chem. Rev. Fett- u. Harz-Industrie*, vol. v. [10], 197-201.)—Reviewing his own method for determining the acetyl value, Kitt's "carbonyl number" method, and the Gregor-Bamberger "methoxyl value" method (ANALYST, 1898, 318), the author expresses the opinion that none of them is capable of replacing the usual acid-, ester-, and saponification-value methods for the identification of, and the detection of adulteration in, the balsams and resins generally. He himself is engaged in perfecting a method based on the circumstance that the saponification-products of the ester-containing resins consist principally of alcohols—*e.g.*, resinotannol, resinol, etc., with merely small quantities of acids; whilst the resins containing only resin acids, yield only acids and no alcohols under the same treatment.

A table is given of the principal physical characteristics of the resins and the contained alcohols and acids. C. S.

Notes on Colophony. R. Schick. (*Zeits. angew. Chem.*, 1899, 27.)—This article is largely argumentative. The author does not agree with Dieterich's idea of determining the "acid number" of resin by allowing it to stand for two hours in the cold with excess of seminormal alcoholic potash, and then titrating back with acid. The value so obtained is simply a number lying between the acid and the saponification values; for if the same process be continued longer, the results are practically the same as when the sample is boiled for twenty-five minutes. In fact, the proportion of cold alkali absorbed rises steadily from the immediate and true acid number during twelve hours until it reaches a maximum agreeing with the hot absorption.

The iodine number of resin varies in different grades between 137 and 173; the process is not complete in six hours, and is of little use in valuing the material. If the colophony be heated for thirty minutes in an open crucible to 300° C. the absorption falls some 44 points. Refractometer numbers, as determined with 20 per cent. solutions of resin in linseed oil at 40° C., range between 71·5 and 92·3, decreasing generally (in contrast with oils) as the iodine number rises. The refraction does not appear very useful in estimating the quality of rosin itself, but is admirably adapted for detecting and estimating its presence when mixed with oils; and the results so obtained agree fairly with the Twitchell process. The practical way of valuing resin depends rather on observations of colour and amount of impurity, such as sand and wood; but the market price depends mainly upon the colour, and therefore fixed grades have a moderately constant tint. Estimation of the matter insoluble in petroleum spirit seems of little use; for there is no evidence that such bodies as are insoluble in that solvent are objectionable to the consumer of the resin. F. H. L.

A Colour Reaction of Lignin. A. Piutti. (*Gazzetta Chim. Ital.*, 1898, xxviii., 168-170.)—The hydrochloride of ortho-bromo-phenetidin,

$$C_6H_3\begin{cases}OC_2H_5(1)\\Br(2)\\NH_2.HCl(4)\end{cases}$$

has the property of staining lignin yellow, while it has no action on cellulose, on ordinary textile fibres, such as linen, silk, cotton or wool, on chitin or on keratin. This reaction appears to be characteristic of lignin, since that substance is stained with the same intensity even after being repeatedly extracted with different solvents. By means of it, it is possible to make an approximate estimation of the amount of wood paste added to a given sample of paper, the colour obtained being compared with those of a standard scale prepared from paper containing known quantities of woody fibre.

Lignin is coloured yellow by the other hydrochloride bodies of similar composition to *o*-Br-phenetidin, such as, for instance, the hydrochlorides *o*-Br-anisidin, of *o*- and *p*-amido-phenol, of *p*-anisidin and of *p*-phenetidin, as well as by other salts of these bases, and the author considers it probable that the reaction is given by amido-phenols generally.

Colorations given by lignin with other classes of compounds have been recorded, as, for example, the violet-rose colour with dimethyl-*p*-phenylene-diamine (Wurster), and with a hydrochloric acid solution of phloroglucinol (Wiesner, *Ding. polyt.*, i., 227 and 397), the yellow colour with the hydrochlorides of naphthylamine and aniline, and the rose colour with pyrrol; but the nature of the coloured compounds formed, or on what group in the lignin molecule the reaction depends, has not yet been determined.

The supposition that the reaction is due to the presence of an aldehydic group in the lignin receives some support from the facts that aldehydes combine very readily with the NH_2 group, and form yellow coloured compounds with amido-phenols.

It is also noteworthy that the violet coloration which phloroglucinol in a hydrochloric acid solution gives with lignin which has been thoroughly extracted with solvents is also obtained with aldehydes. C. A. M

The Examination of Caoutchouc Milk. Girard and Lindet. (*Bull. Soc. Chim.*, 1898, xix., 812-815.)—In whatever manner the incision is made in the bark of the tree, the caoutchouc milk has always the same appearance, being an opaque liquid with a great resemblance to animal milk. Under the microscope it appears as an emulsion of innumerable fine globules, from 3 to 5 μ in diameter, in a "serum." As a rule the density of the milk is less than unity, with the exception of that from different species of the Algerian *ficus*. The density of the "serum" freed from caoutchouc varies from 1·037 to 1·040.

In order to determine the percentage of caoutchouc in the liquid, Girard modified the nature of the "serum" by the addition of 95 per cent. alcohol, so that the globules collected on the surface and became united. The alcohol was added little by little, with constant stirring, until, when a volume about equal to that of the milk had been added, the whole of the rubber collected on the surface, leaving below a limpid, amber-coloured liquid.

The following table gives the percentage of caoutchouc thus obtained from different caoutchouc milks :

	Density.	Per cent. Caoutchouc.
Milk from Pernambuco (*Haucornia*)	0·990	31·6
Africa	0·987	33·4
Para (*Siphonia elastica*)	0·986	42·6
Nicaragua (*Castilloa elastica*)	0·980	32·3
Algiers (*Ficus macrophylla*)	1·000	37·5
,, ,, ,, ...	1·005	37·1
,, (*F. elastica*)	1·001	17·3
,, (*F. nitida*)	0·971	31·3
,, (*F. lævigata*)	1·005	28·0
New California (*Kickxia Africana*) ...	1·005	27·0

This similarity between caoutchouc milk and animal milk suggested to Girard the possibility of extracting the caoutchouc by churning the liquid previously heated to 50° C. This was successful on a small scale in the laboratory, and was then tried on a large scale on freshly collected milk in various parts of French Guiana by Bouery, who, after two years' experience, reported that the results were satisfactory. C. A. M.

The Oxy-ferments of Milk and of Saliva. R. Dupouy. (*Journ. Pharm. Chim.*, 1898, 551-553.)—In a former communication the author described the variation in the nature of different milks as regards the activity of the oxydases which they contain, and showed that they can be classified into two groups—those which contain an active oxydase, and those in which (like human milk) the oxy-ferment is very weak.

Carnot (ANALYST, xxiii., 80) discovered the presence of an oxydase in saliva, but, as the author obtained negative results on repeating the experiments, he has made a further investigation of the subject, using exclusively a 1 per cent. solution of guaiacol as his reagent, this being very stable as compared with Carnot's reagents (tincture of guaiacum, solution of paraphenyl-diamine, 1 : 1,000, and paraphenyl-diamine with naphthol in alkaline solution).

The presence of an oxydase in saliva can be demonstrated by adding an equal volume of the guaiacol solution and a drop of a solution of commercial hydrogen dioxide diluted with nine volumes of water. There is an immediate brown coloration, becoming more intense, and finally disappearing after a considerable time. On warming the saliva, the coloration with guaiacol appears readily until the temperature reaches 92° C ; above that temperature the colour appears very slowly, and is hardly perceptible. The author therefore regards 92° C. as the temperature at which the activity of the oxydase is destroyed. It is also rendered completely inactive by an addition of a mineral acid, such as, for instance, a 0·2 per cent. solution of hydrochloric acid. C. A. M.

INORGANIC ANALYSIS.

Notes on the Electrolytic Determination of Iron. S. Avery and B. Dales. (*Ber.*, 1899, xxxii., 64.)—Three processes were investigated : that recommended by Classen, in which the electrolyte is ammonium oxalate ; Smith's method with acid sodium citrate ; and Moore's with ammonium metaphosphate. Classen's gives a

deposit of iron containing between 0·15 and 0·5 (mean 0·22) per cent. of carbon, and about the same amount of iron remains in solution. Smith's iron contains between 1·2 and 5·0 per cent. of carbon. Moore's metal shows an excess in weight of 0·2 to 0·5 per cent., and the impurity consists wholly or in part of phosphorus and carbon. To ascertain whether carbon was the sole foreign ingredient in the metal thrown down from an oxalate bath, some of the iron was burnt in a current of air, passing the gas into baryta water; the amount of carbon calculated from the carbon dioxide was from 0·21 to 0·42 per cent. of the metal examined. The by-product in the Moore process is a heavy black substance, soluble in hot strong acids, but insoluble in dilute ones. F. H. L.

Impure Iron: A Possible Source of Error in Ulsch Nitrogen Estimations. L. Brandt. (*Chem. Zeit.*, 1899, xxiii., 23.)—When a blank experiment was recently carried out to check the purity of the various reagents used in estimating nitrogen in Chili saltpetre by the Ulsch process, it was found that the acid distillate required too little caustic soda for neutralization—the error amounting to 0·8 per cent. of nitrogen on the supposed sample. The impurity was traced to the iron, which evolved ammonia when it was dissolved in acid and boiled with caustic soda, but did not itself contain ammoniacal nitrogen. Boiled with alkali alone, nitrogen could not be detected in the filtrate either as nitrate, nitrite, or cyanide; but after heating with metallic potassium, cyanide was discovered. By ignition in hydrogen the impurity was removed; whereas neither extraction with water, alcohol, nor ether affected it. F. H. L.

Volumetric Estimation of Alkalis and Acids by means of Iodine. C. F. Walker and D. N. H. Gillespie. (*Zeits. anorg. Chem.*, 1899, xix., 194.)—When iodine acts upon a solution of a metallic hydroxide at a temperature high enough to destroy any trace of hypoiodite, a perfectly neutral liquid is produced which contains 1 molecule of iodate to 5 of iodide. On adding dilute acid, these two salts interact in the well-known way, evolving 6 atoms of iodine; and by titration with thiosulphate or arsenious acid, the iodine—that is to say, the original hydroxide—may be estimated. Similarly, an acid may be neutralized by a known excess of alkali standardized as aforesaid, when determination of the surplus will give the strength of the acid. The process has been tested on the hydroxides of the alkalis and alkaline earths, on sulphuric and hydrochloric acids; and although the precautions necessary to avoid loss of iodine and carbonation of the liquid perhaps render it somewhat complicated, the reaction proceeds so smoothly that it should be serviceable for the indirect analysis of acids and probably for other suitable compounds. It cannot, however, be employed on alkali-metal carbonates. The method outlined by Phelps (ANALYST, 1897, xxii., 55) may with advantage be slightly modified. A moderate excess of decinormal iodine is placed in a lightly-covered Erlenmeyer flask, the alkali is added (or, in determining acid, the acid is added, followed by a measured excess of standard alkali), and the whole is boiled till all free iodine is volatilized. The bulk of the liquid in all tests should be uniform and as small as possible, starting with about 100 c.c. and boiling down to about 35 c.c. The vessel is cooled in a

stream of water, 10 c.c. of 1 : 8 sulphuric acid (or hydrochloric acid, if barium be the metal) are introduced, and the liquid titrated with thiosulphate and starch. Blank experiments show that the error involved by conducting this titration in an acid solution is not apparent under the particular conditions referred to. F. H. L.

Some Applications of Hydrogen Peroxide in Quantitative Analysis. P. H. Walker. (*Jour. Amer. Chem. Soc.*, 1898, xx., 513-515.)—*The Separation of Titanium from Iron.*—The cold slightly acid solution of the mixed sulphates (about 150 c.c.) is mixed with 100 c.c. of a solution of hydrogen peroxide (about 2·5 per cent.), and slowly run into a mixture of 100 c.c. of hydrogen peroxide and 30 c.c. of strong ammonia, with constant stirring. The precipitated ferric hydroxide is filtered off at once, and washed with cold dilute ammonia containing a little hydrogen peroxide. The precipitate is dissolved off the paper with a mixture of hydrochloric acid and hydrogen peroxide, again treated with an excess of hydrogen peroxide, and reprecipitated as before. This treatment is repeated a third time, after which the ferric hydroxide can be dissolved, and the iron determined in the usual way.

The filtrates containing the titanium are boiled down, and the titanium hydroxide ($Ti(OH)_4$) which precipitates is filtered off, washed with hot ammonium nitrate solution, ignited, and weighed as TiO_2.

An experimental mixture consisting of titanium dioxide fused with potassium bisulphate, and mixed with iron ammonium alum, gave the following results when analysed by this method :—

	Gramme.
Titanium oxide found by first separation	0·0872
,, ,, second ,, ...	0·0104
,, ,, third ,, ...	0·0041
Total found	0·1017
Titanium oxide taken	0·1016

The Separation of Uranium from Iron.—The presence of a large excess of hydrogen peroxide prevents the precipitation of uranium by sodium hydroxide, but does not interfere with the precipitation of the iron. A slightly acid solution of the mixed salts is mixed with 50 c.c. of hydrogen peroxide, and run slowly, with constant stirring, into a solution of 5 grammes of sodium hydroxide in 50 c.c. of water, mixed with 50 c.c. of hydrogen peroxide. The liquid is diluted to 400 c.c. with hot water, and the iron filtered off and washed. The filtrate (which contains all the uranium) is acidified with hydrochloric acid, evaporated to dryness on the water-bath, the residue heated for an hour at 110° C. to separate any silica, dissolved in hydrochloric acid, filtered, and the uranium precipitated with ammonia, washed with ammonia, ammonium nitrate solution and water, dried, ignited, and weighed as U_3O_8.

In a solution containing about equal weights of Fe_2O_3 and U_3O_8 the following amounts of uranium oxide were found :

	U_3O_8 Taken.	U_3O_8 Found.	Difference.
(*a*)	0·1129	0·1124	−0·0005
(*b*)	0·1056	0·1064	+0·0008

The Separation of Zirconium from Uranium.—The solution of the mixed sulphates is mixed with 50 c.c. of hydrogen peroxide, and run into a solution containing 5 grammes of sodium hydroxide in 50 c.c. of water, and 50 c.c. of hydrogen peroxide. No precipitate forms at first, but after heating on the water-bath for thirty minutes the zirconium oxide settles down as a heavy precipitate. This is collected, washed, and dissolved in a warm mixture of hydrochloric acid and hydrogen peroxide. The solution is boiled, the zirconium precipitated with ammonia, and the precipitate washed with ammonium nitrate solution and with water, dried, ignited, and weighed as ZrO_2.

Zirconium Dioxide Taken.	Zirconium Dioxide Found.	Difference.
0·2150	0·2158	+0·0008

C. A. M.

Recognition of Ozone in Presence of Nitrous Acid and Hydrogen Peroxide. G. Erlwein and T. Weyl. (*Ber.*, 1898, xxxi., 3158.)—An alkaline solution of the hydrochloride of metaphenylenediamine, which is naturally colourless, is rapidly changed to wine-red by ozone; whereas nitrites and hydrogen peroxide do not affect it, and oxygen only causes a faint coloration after the lapse of some time. The ortho- and para-diamines behave similarly, but the meta-compound is the most useful. A solution containing 0·1 or 0·2 gramme of the hydrochloride in 90 c.c. of water and 10 c.c. of 5 per cent. caustic soda should be freshly prepared each time it is wanted; 25 c.c. of the reagent are tinted yellowish-brown in 5 seconds by 0·08 milligramme of ozone. Conversely, liquids containing 6 milligrammes of diamine per litre of faintly alkaline water are yellowed by ozone, excess of gas destroying the colour. Twenty-five milligrammes of diamine yield a yellowish brown. Strong sulphuric acid changes the colour to a very dark red; nascent hydrogen (zinc and acid or alkali) bleaches it, but on agitation with air it returns to its original appearance.

F. H. L.

Preparation of Starch-Zinc Iodide Solution. A. Seyda. (*Chem. Zeit.*, 1898, xxii., 1086.)—Two or three grammes of potato-starch are rubbed down most thoroughly with 50 or 100 c.c. of water, allowed to stand for one hour under cover, then heated in a strong flask for two to four hours at a temperature of 130° C. in a bath of rape oil. The liquid is diluted to about 750 c.c. with hot water, shaken, set aside for twenty-four hours, and filtered. Twenty grammes of zinc chloride and 2 grammes of zinc iodide (made from zinc dust and iodine) are introduced; if necessary a few drops of centinormal thiosulphate are run in to destroy any blue colour, the whole is made up to 1 litre, well mixed, and finally filtered after twenty-four hours' standing into a bottle of brown glass. The solution keeps indefinitely.

F. H. L.

List of Atomic Weights for Analytical Purposes. (*Chem. Zeit.*, 1898, xxii., 1031.)—A commission, consisting of Professors Landolt, Ostwald, and Seubert, appointed jointly by the German Chemical Society and the German "Reichs-

gesundheitsamt," has recently drawn up the following list of atomic weights, which it is hoped will be uniformly adopted for all commercial analytical purposes throughout Germany. For the sake of greater convenience, the table has been based on the unit O = 16·00, and the figures are given to as many places of decimals (if any) as may insure the last being free from error. The weights of those "elements" marked with a point of interrogation are included under reserve.

TABLE OF ATOMIC WEIGHTS.

	Symbol.	Atomic Weight.		Symbol.	Atomic Weight.
Aluminium	Al	27·1	Neodymium (?)	Nd	144
Antimony	Sb	120	Nickel	Ni	58·7
Argon (?)	A	40	Niobium	Nb	94
Arsenic	As	75	Nitrogen	N	14·04
Barium	Ba	137·4	Osmium	Os	191
Beryllium	Be	9·1	Oxygen	O	16·00
Bismuth	Bi	208·5	Palladium	Pd	106
Boron	B	11	Phosphorus	P	31·0
Bromine	Br	79·96	Platinum	Pt	194·8
Cadmium	Cd	112	Potassium	K	39·15
Cæsium	Cs	133	Praseodymium (?)	Pr	140
Calcium	Ca	40	Rhodium	Rh	103·0
Carbon	C	12·00	Rubidium	Rb	85·4
Cerium	Ce	140	Ruthenium	Ru	101·7
Chlorine	Cl	35·45	Samarium (?)	Sa	150
Chromium	Cr	52·1	Scandium	Sc	44·1
Cobalt	Co	59	Selenium	Se	79·1
Copper	Cu	63·6	Silicon	Si	28·4
Erbium (?)	Er	166	Silver	Ag	107·93
Fluorine	F	19	Sodium	Na	23·05
Gallium	Ga	70	Strontium	Sr	87·6
Germanium	Ge	72	Sulphur	S	32·06
Gold	Au	197·2	Tantalum	Ta	183
Helium (?)	He	4	Tellurium	Te	127
Hydrogen	H	1·01	Thallium	Tl	204·1
Indium	In	114	Thorium	Th	232
Iodine	I	126·85	Tin	Sn	118·5
Iridium	Ir	193·0	Titanium	Ti	48·1
Iron	Fe	56·0	Tungsten	W	184
Lanthanum	La	138	Uranium	U	239·5
Lead	Pb	206·9	Vanadium	V	51·2
Lithium	Li	7·03	Ytterbium	Yb	173
Magnesium	Mg	24·36	Yttrium	Y	89
Manganese	Mn	55·0	Zinc	Zn	65·4
Mercury	Hg	200·3	Zirconium	Zr	90·6
Molybdenum	Mo	96·0			

F. H. L.

A Soda Glass Unsuitable for Chemical Apparatus. E. Hoyer. (*Chem. Zeit.*, 1898, xxii., 1033.)—Liebermann has already called attention to the fact that some chemical apparatus now on the market is made of such strongly alkaline glass that it is quite unfit for analytical purposes. A specimen of this material contained 66·52

per cent. of SiO_2, 24·52 Na_2O, 7·81 CaO, and 0·48 Fe_2O_3 and Al_2O_3. On exposure to air in fine powder it gained 0·8 per cent. in weight in five weeks, and decreased very slightly when heated to 115° C. Treated even with cold water for twenty-four hours it lost 0·56 per cent. by weight or 2·3 per cent. of its content of alkali; on boiling with water and cold or hot extraction with hydrochloric acid it lost more severely eight hours' boiling with pure hot water causing a diminution of 11·80 per cent. in the total weight of the powder. F. H. L.

APPARATUS.

An Apparatus for Determining Melting-Points. L. N. Vandevyver. (*Ann de Chim. Anal. Appl.*, 1898, iii., 397-399.)—After describing the principal methods used for determining the melting-points of substances, and pointing out the restrictions of each, the author describes an apparatus which he considers to be of more general applicability.

He takes as the melting-point that temperature at which a substance in passing from the solid to the liquid state leaves a sign of the change on an object with which it is in contact.

His apparatus consists of a rod, A B (Fig. 1), to which is attached a fixed ring C, and a movable ring, D, which can slide down on to C. A piece of white filter paper is fixed between the two rings, and a fragment of the substance of which the melting-point is to be determined is placed upon this. The rod terminates at B in a small mirror, M, placed at an angle of 135°.



Fig. 1.

The upper end of the rod is fixed in a cork which fits into a large test-tube, and a thermometer, T, is placed with its bulb close to the substance. The test-tube is immersed in a beaker, V (Fig. 2), containing water, glycerin, or paraffin, etc., which is kept in motion during the warming by means of the agitator, R. When water is used it is advisable to have a small brush, S, attached to the end of R to dispel the air-bubbles which become attached to the glass and obscure the view.

The moment the substance fuses a stain is produced on the paper, and this is reflected by the mirror. The author states that he has determined the melting-points of a large number of substances by this method, and has obtained a greater agreement in duplicate determinations than was possible by any other process.

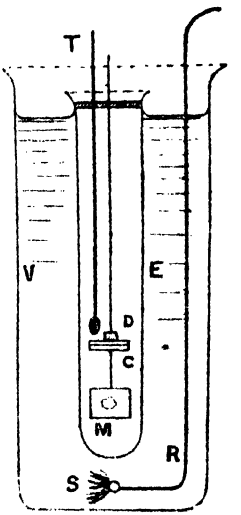


Fig. 2.

When a substance is being examined which stains paper in the solid state, a piece of ground or polished glass is substituted for the paper, and in this case also the change in the state of the substance is clearly shown in the mirror. For high temperatures the ordinary mirror is replaced by a metallic mirror. C. A. M.

THE ANALYST.

APRIL, 1899.

PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS.

THE monthly meeting of the Society was held on Wednesday evening, March 1, in the Chemical Society's Rooms, Burlington House, the President, Mr. W. W. Fisher, M.A., occupying the chair.

The minutes of the previous meeting were read and confirmed.

Certificates of proposal for election to membership in favour of the following candidates were read for the second time: Percy T. Adams, assistant to Dr. Matthew A. Adams, Maidstone; Arthur French Angell, assistant to Mr. Arthur Angell, Southampton; John B. Ashworth, assistant to Mr. Alfred Smetham, Liverpool; Alfred Joseph Bull, assistant to Mr. Wm. Chattaway, and senior assistant in the Physical Laboratory, Birkbeck Institution, London; Charles Crocker, assistant to Messrs. Morgan, Son and Seyler, Swansea; John S. Ford, analytical chemist, Edinburgh; C. A. Hackman, A.I.C., assistant to Mr. Alfred C. Chapman, London; Norman Leonard, B.Sc., F.I.C., assistant to Dr. Stevenson, Guy's Hospital, London; J. F. Liverseege, F.I.C., assistant analyst to the Corporation of Birmingham; Richard Murray, chemist to Messrs. Brotherton and Co., Leeds; G. E. Scott-Smith, F.I.C., assistant to Mr. Alfred H. Allen, Sheffield; W. P. Skertchly, assistant to Mr. Otto Hehner, London; Harry Metcalfe Smith, F.I.C., assistant to Dr. Stevenson, Guy's Hospital, London; Lionel W. Stansell, assistant to Dr. Matthew A. Adams, Maidstone; and W. Northfield Yarrow, assistant to Mr. A. W. Stokes, London; and in favour of the following candidates for the first time: Julian L. Baker, F.I.C., chief chemist to the Beetroot Sugar Association, London; Herbert Edward Burgess, chemist to the London Essence Co., London; Louis Charles Deverell, chief assistant to Mr. Benedict Kitto, London; Julius Lewkowitsch, analytical and consulting chemist, London; F. R. O'Shaughnessy, Associate R.C.S., analytical chemist, London; Arthur Marshall, A.I.C., analytical chemist and assayer, London; and Edward T. Shelbourn, A.I.C., assistant analyst to the London County Council, London.

The following papers were read: "Some Analyses of Ginger," by E. G. Clayton; "Note on Boric Acid in Milk Samples," by E. G. Clayton; "Caper Tea," by John White; and a "Note on a Possible Source of Error in Modifications of the Leffmann and Beam Method of Fat Estimation in Milk," by H. Droop Richmond and F. R. O'Shaughnessy.

Mr. Wm. Chattaway exhibited an apparatus for collecting samples of water in Winchester quart bottles at given depths.

A NEW TEST FOR FORMALDEHYDE.

By Norman Leonard, B.Sc., F.I.C., and H. Metcalfe Smith, F.I.C.

(*Read at the Meeting, January* 4, 1899.)

In the determination of the fat "by the Werner-Schmid process" in milk containing formaldehyde, we have occasionally noticed the production of a violet colour when the milk was heated with concentrated hydrochloric acid. This behaviour of formalized milk has also been observed by Fisher (Analyst, xx., 156), whilst other experimenters have noted the production of a *yellow* colour (*loc. cit.*, and Analyst, xxi., 97). Further study of this reaction has enabled us to explain these discrepancies, and to show that it forms a most delicate test for formaldehyde in milk.

The certain production of the violet colour requires the use of a larger proportion of hydrochloric acid than is employed in the Werner-Schmid process. In the case of milks containing such quantities of formaldehyde as are commonly added, a little of the sample should be heated gently in a test-tube with from three to five times its volume of concentrated hydrochloric acid. Under these conditions, a fine violet colour quickly appears, even if the milk contains so little as 0·0001 per cent. (1 per 1,000,000) of formaldehyde, whilst if much over 0·1 per cent. is present a *yellow* colour is produced. In the latter case, however, the violet colour is readily obtained if a large excess of hydrochloric acid is used, so as to sufficiently dilute the liquid. So far as we have been able to ascertain, the reaction is not given by other aldehydes, or by any substance but formaldehyde. We may add that the reaction takes place, though less quickly, at the ordinary temperature, and by working in this way any confusion arising from the action of the acid on the milk itself is entirely avoided.

Bearing in mind the fact, pointed out by one of us (Analyst, xxi., 157), that the presence of a trace of some oxidizing agent is an essential condition in Hehner's test for formaldehyde, experiments were made to ascertain whether such was the case in the reaction described above. The "pure redistilled" hydrochloric acid used by us certainly contained a minute trace of a ferric salt, and gave a slight blue coloration when largely diluted and added to a solution of cadmium iodide and starch paste. When, however, the acid was digested for a short time with a little metallic zinc and then poured off, it gave these reactions no longer, and had also lost the power of producing the violet colour when heated with formalized milk. On adding a trace of ferric chloride, or of bromine, the reaction was readily obtained. It seems probable, therefore, that the coloured products formed in Hehner's test, and in the reaction we have described, are one and the same, the acid employed acting, perhaps, merely as a dehydrating agent.

Discussion.

The President (Dr. Dyer) having invited discussion:

Mr. W. W. Fisher said he remembered noticing, some years previously, in testing a sample of milk somewhat liberally treated with formaldehyde, that a colour was produced on boiling with hydrochloric acid. He did not investigate the cause of the reaction, but he now had a suspicion that it might not be due to formaldehyde only, as milk heavily treated with boracic acid showed a similar colour reaction.

When milk was treated with either formaldehyde or boracic acid, the curd was obtained in such a condition as to be very insoluble in hydrochloric acid, a fact which pointed to some change in the constitution of the casein itself, produced by the addition of the antiseptic; and he thought it possible that the colour reaction referred to might arise from an alteration of the casein, and not simply from the presence of formaldehyde.

Dr. RIDEAL thought that the presence of milk would be essential to the colour being obtained. Mr. Hehner, when describing his test with sulphuric acid, had noted that formaldehyde alone gave no reaction with sulphuric acid. Fritzmann (*Chem. Centr.*, 1898, i., 218) has shown that the blue colour obtained with milk and sulphuric acid is due to the presence of an oxidizing agent, usually *nitric* acid or nitrates in the milk or acid used.

Mr. LEO TAYLOR remarked that when formalin had been added to milk which was being tested by the Leffmann-Beam or by the Werner-Schmid process, the casein was always found to be difficult to dissolve.

Mr. A. E. EKINS confirmed Mr. Taylor's experience with regard to the insoluble condition of milk to which formalin had been added, as observed in the working of the Leffmann-Beam process.

ADULTERATION OF SPIRITS OF NITRE WITH POTASSIUM NITRATE.

BY W. F. LOWE, A.R.S.M., F.I.C.

(*Read at the Meeting, January* 4, 1899.)

THE following short note on what appears to me to be an unusual form of adulteration of spirits of nitre will, I think, be of interest to the Society, as such a form of adulteration does not appear to have been recorded.

A sample of "spirits of nitre" was received in the usual way from one of the food inspectors of my district. On examination I found that it yielded no gas whatever in the nitrometer when treated with potassium iodide and sulphuric acid, but with ferrous sulphate and sulphuric acid it gave a faint brown ring. The specific gravity at 15·5° C. was 0·9073. I noticed a whitish deposit on the pipette used for transferring some of it to the nitrometer, and this led me to examine the sample for solid matter, when I found it to contain 1·12 per cent. of potassium nitrate.

I have since had several similar samples, so that this was not an isolated case, and apparently a good deal of this stuff has been made up for sale, judging from the printed label which was put in (but unsuccessfully) by the defence when one of the cases was heard in court:

"SPIRITUOUS SOLUTION OF NITRE.

"N.B.—This is not the ordinary Sweet Spirit of Nitre, and must not be sold as such; for most purposes it is a much superior article. It contains an ingredient not in the Nitre of the Pharmacopœia, making it much more effective on the Skin and Kidneys."

In spite of the label, I think it must certainly be considered a gross adulteration, as it is undoubtedly usually sold for spirits of nitre.

Mr. JOHN WHITE said that he had been told by a firm of wholesale druggists that such an article as this was actually prepared and put on the market, and since receiving that information he had always determined the total solids in samples of sweet spirit of nitre that he had to examine, but he had never met with any such sample as that referred to by Mr. Lowe.

ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

FOODS AND DRUGS ANALYSIS.

The Detection of Renovated Butter. C. B. Cochran. (*Journ. Franklin. Instit.*, 1899, cxlvii., 94-97.)—Renovated or "process" butter is prepared from stale and unsaleable butter by melting out the fat, and either simply cooling it rapidly by means of ice, or (less frequently) mixing it with milk or butter-milk, and again churning it into butter.

In order to distinguish this product from fresh butter, the author recommends the following tests: (1) Microscopic examination of the sample by means of polarized light both with and without a selenite plate. (2) Valenta test. (3) Jean's modification of the valenta test. (4) Determination of free fatty acids. (5) Reichert value.

When examined under the microscope with polarized light with a selenite plate, fresh butter shows a uniformly coloured field in any position of the analyzer, whilst the field of renovated butter is mottled and of various colours. Without the selenite plate, renovated butter gives a dark field with white patches; pure butter, a uniformly dark field. This difference is due to the state of incipient crystallization of the rapidly-cooled fat. When fresh butter is melted and allowed to cool slowly, the appearance is quite distinct from that of renovated butter, the large crystalline masses being globular in form and showing a distinct cross by polarized light.

Old butter or butter which has been exposed to changes of temperature, sometimes has a similar appearance under the microscope to that of renovated butter; but although the author has obtained the variegated field with polarized light in the case of such butters, when using the selenite plate, he has never seen the white patches which renovated butter shows without the selenite plate. In his opinion, ordinary butter does not readily assume the same crystalline form as "process" butter. The crystalline structure of oleomargarine is much more pronounced than that of renovated butter.

The turbidity temperature of renovated butter in the Valenta test is lower than that of fresh butter, and more acetic acid is dissolved by it in Jean's modification of the test. The percentage of free fatty acid is also usually high.

The following table gives the results of the examination of three samples of such butter and of a sample of fresh butter. Sample 4 was known to be renovated, and samples 2 and 3 judged to be so by the results of the analysis.

Sample.	Microscopic Appearance.	Valenta Test. Acetic Acid 98·7 per Cent.	Per Cent. of Acetic Acid dissolved.	N-Alkali required to neutralize 100 Grammes of Fat.
1. Fresh Butter	not crystalline	60° C.	70	1·1 c.c.
2. Renovated Butter	crystalline	48·5° C.	100	6·5 c.c.
3. ,, ,,	,,	50° C.	100	9·4 c.c.
4. ,, ,,	,,	49° C.	100	8·1 c.c.

The author also calls attention to the characteristic form in which butter crystallizes from amyl alcohol. This is quite distinct from the form of oleomargarine or lard crystals, and has enabled him in many cases to detect the presence of a small quantity of foreign fat in butter. C. A. M.

The Determination of Added Starch in Pressed Yeast. F. Dewalque. (*Bull. de l'Ass. belge*, 1898, xii., 264-269.)—The only method by which the author has been able to obtain good results is that of Maercker. Ten grammes of the yeast are mixed with 100 c.c. of water, and heated for twenty minutes at 60° to 70° C. The temperature is then brought to 60° C., ten c.c. of malt extract (prepared by extracting 100 grammes of green malt with 500 c.c. of water) added, and the temperature kept constant for half an hour. The saccharified liquid is brought to 250 c.c., filtered, 200 c.c. of the filtrate inverted with 15 c.c. of hydrochloric acid (specfic gravity 1·125), and diluted to 500 c.c. The reducing sugar in 25 c.c. of this is determined with Fehling's solution in the usual manner, and a correction made for the amount of copper reduced by the malt extract added.

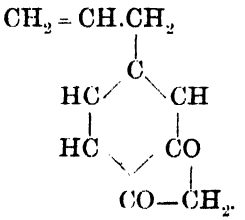
The author prefers to calculate the quantity of starch on normal yeast, which, according to Hayduck, contains 74 per cent. of water, although he considers this estimate of the water too high. C. A. M.

Notes on Cazeneuve's Method of Detecting Artificial Colour in Wines. E. Comboni. (*Staz. Speriment. Agrar. Ital.*, 1898, xxxi., 490-498.)—Cazeneuve's method consists in shaking 10 c.c. of the wine with 20 centigrammes of yellow mercuric oxide and filtering, the filtrate being colourless (according to Cazeneuve) when the wine is pure. The author has made experiments with this test on different kinds of wine, from which he finds that this quantity of mercuric oxide is frequently insufficient to precipitate all the natural colouring matter, and that more certain results can be obtained by using 1·5 gramme to 10 c.c. of wine. But when the wine is of American origin or has been mixed with American wine the test is useless, and the colouring matter is not removed by the addition of as much as 8 grammes of the reagent to 10 c.c. It is preferable to use the mercuric oxide in the freshly precipitated moist condition, and to add it to the cold wine.

Treatment of the wine with amylic alcohol saturated with barium hydroxide also shows this difference between Italian and American wines, the filtrate from the former being colourless, while that from the latter is invariably rose-coloured. The author considers that the method of König and Arata (no reference given), in which

the wine is treated with fat-free wool, is the only certain test in dealing with American wine. C. A. M.

The Composition and Characteristics of Sassafras Oil. C. Kleber. (*Amer. Journ. Pharm.*, 1899, lxxi., 27-32.)—Commercial oil of sassafras is distilled exclusively from the sassafras roots, and chiefly from the bark of the root. At first it is nearly colourless, but on exposure to light and air gradually changes to yellow or brown. Its specific gravity lies between 1·07 and 1·08, and its optical rotation varies from +3° to +4°. On exposure to cold crystals of saffrol gradually deposit, and by repeated treatment in a freezing mixture about 80 per cent. of that substance can be isolated. Pure saffrol is an optically inactive colourless liquid, freezing at 8° C. and boiling at 232° C. Its formula is $C_{10}H_{10}O_2$, and it appears to be the methylene ester of an allyl catechol.



The parts of sassafras oil which remain liquid even in a freezing mixture can be separated by fractional distillation into a fraction boiling between 155° and 175°, and consisting chiefly of pinene, $C_{10}H_{16}$, with a small amount of another terpene, phellandrene, $C_{10}H_{16}$. The higher fractions contain about 0·5 per cent. of a substance which can be extracted by dilute alkali, and consists of eugenol $C_{10}H_{12}O_2$.

The fractions boiling about 200° C., yield, on cooling, colourless prisms of ordinary dextro-camphor, $C_{10}H_{16}O$, while the highest fractions contain a small quantity of a sesquiterpene $C_{15}H_{24}$ which, from its colour reactions, the author believes to be cadinene.

The percentage composition of oil of sassafras bark may thus be summarized: safrol, 80; pinene and phellandrene, 10; *d*-camphor, 6·8; eugenol, 0·5; cadinene (?); and residue, 2·7.

The resemblance between oil of sassafras and oil of camphor has led to manufacturers substituting the oily by-products of the manufacture of camphor for sassafras. According to the author, many of the commercial sassafras oils are fractions of Japanese camphor oil of the same gravity (1·07) as the natural oil. They are largely used, especially by soap manufacturers, on account of their lower price.

The oil of sassafras-leaves, which is not a commercial product, differs greatly from the root oil. The yield obtained by steam distillation amounts to about 0·028 per cent. of the weight of the fresh leaves. The oil when fresh is greenish yellow, and turns to reddish brown on keeping. Its specific gravity is 0·873, its optical rotation + 6·25, and it has an agreeable odour recalling that of oil of lemon and oil of citronella. It contains a considerable amount of citral, $C_{10}H_{16}O$, of geraniol, $C_{10}H_{18}O$, and of the isomeric linalol, the acetic and valerianic esters of the two latter alcohols,

and several terpenes, including pinene and phellandrene. In the highest boiling parts of the oil some cadinene also appears to be present, and a paraffin-like substance melting at 58° C. (*Cf.* ANALYST, xxiii., 212.) C. A. M.

Valuation of Phosphorated Oil. H. Ekroos. (*Arch Pharm.*, 1898, 627; through *Deutsche Chem. Zeit.*, 1899, xiv., 27.)—The author has unsuccessfully attempted to determine the proportion of elemental phosphorus in its solution in almond oil, which is known as *Oleum phosphoratum*. He tried treatment with bromine and iodine, followed by water, in order to obtain phosphoric acid; also the formation of silver phosphide by the aid of silver nitrate; also distillation of the phosphorus in carbon dioxide. None of the processes was accurate; and it would appear that, especially on keeping, the phosphorus enters into combination with the fatty acids of the oil, and is no longer amenable to ordinary analytical separations.

F. H. L.

The Colour-Reactions of Heroïne. G. Wesenberg. (*Journ. Pharm. Chim*, 1899, ix., 16-18.)—Heroïne is the diacetic ester of morphine—

$$C_{17}H_{17}\begin{matrix}OOC.CH_3\\OOC.CH_3\end{matrix}NO,$$

and is prescribed in place of codeïne phosphate. It is precipitated by the usual alkaloidal reagents, the most sensitive being iodine in potassium iodide, which causes a turbidity in a solution containing 1 part in 100,000. The author gives the following table of its colour-reactions in comparison with those of morphine and codeïne:

Reagent.	Morphine.	Codeïne.	Heroïne.
Concentrated H_2SO_4 ...	Nil	Nil	Nil
H_2SO_4 + 1 drop of HNO_3 ...	Blood-red	Blood-red	Yellow, becoming blood-red on warming
H_2SO_4 + 1 drop of ferric chloride in 100 c.c. On warming	Greenish-blue	Pure blue	Greenish-blue
Potassium ferricyanide + ferric chloride	Immediate blue precipitate	Nothing at first; blue coloration after some time	Like codeïne
Nitric acid	Blood-red, becoming yellow	Yellowish-red	Yellow, becoming red on warming
Iodic acid	Separation of iodine	Nil	Nil

C. A. M.

ORGANIC ANALYSIS.

Estimation of Formaldehyde. O. Blank and H. Finkenbeiner. (*Ber.*, 1898, xxxi., 2979.)—The authors find that Legler's method of titrating formaldehyde with ammonia gives results some 1·5 per. cent. below other methods of estimation (*cf.* ANALYST, 1897, xxii., 221). The following process for oxidizing it to formic acid by means of hydrogen peroxide in the presence of an alkali is preferable and quite accurate: 3 grammes of the solution or 1 gramme of the solid are introduced into 25 c.c. of 2N sodium hydroxide solution contained in a tall flask; 50 c.c. of 2·5 or 3·0 per cent. pure hydrogen peroxide are then cautiously and slowly dropped in, the addition lasting about three minutes. After standing from two to three minutes longer, the alkali is titrated with 2N sulphuric acid and litmus. In the case of formalins stronger than 45 per cent. 30 c.c. of sodium hydroxide solution should be used; samples weaker than 30 per cent. should be allowed to stand ten minutes before titration. The volume of standard alkali used, multiplied by six, gives the formaldehyde in 1 gramme of the solid, or multiplied by two in 3 grammes of the solution.

A similar reaction occurs, but much more slowly, with benzaldehyde, especially in presence of a trace of ferrous sulphate; acetaldehyde is also oxidized, but it is doubtful whether the decomposition is complete. Paraldehyde is very slowly attacked, and even ferrous salts do not greatly quicken the action. F. H. L.

Note on Testing Formaldehyde. L. F. Kebler. (*Amer. Journ. Pharm.*, 1898, lxx., 432-433.)—In estimating the strength of a solution of formaldehyde by Legler's method (*Berichte*, xvi., 1333), in which the formaldehyde is shaken with a standard solution of ammonia and the uncombined ammonia distilled over and titrated with standard acid, the author finds that, as a rule, the reaction between the aldehyde and ammonia is rather slow, and that if they are left in contact for less than six hours, the results will probably be considerably too low. Thus, a commercial sample of formalin showed a strength of 16 per cent. when the ammonia was distilled after a fifteen minutes' reaction, but by increasing the time of contact the percentage gradually increased up to 37·5 per cent. after six hours. This result was also obtained when the mixed solutions were left overnight. The results obtained at different times under six hours were not constant in duplicate determinations of the same sample.

C. E. Smith, who was delegated by the Pharmacopœial Research Committee to investigate the efficiency of various methods proposed for the determination of formaldehyde, found that by making a suitable correction the strength could be estimated by leaving it for about fifteen minutes in contact with normal ammonia solution (*Amer. Journ. Pharm.*, lxx., 86). The author's explanation of Smith's obtaining such results is that the number of samples of formalin examined by him was too limited, and evidently not representative of the commercial article.

C. A. M.

A New Method of Detecting and Estimating Acetone. G. Denigès. (*Journ. Pharm. Chim.*, 1899, ix., 7-14.)—In a recent communication (*Comptes Rend.*,

1898, cxxvi., 1868) the author described certain insoluble or but slightly soluble compounds which mercuric sulphate forms with acetones of the fatty series. When dried at a temperature not exceeding 100° C. those compounds which are formed without secondary reactions have a composition corresponding to the formula $[(SO_4Hg)_2.3HgO]_3.4[CO.R_2]$.

The readiness with which ordinary acetone forms the insoluble compound, $[(SO_4Hg)_2.3HgO]_3 4[CO.(CH_3)_2]$, and the high molecular weight of that compound (3,952) as compared with that of the acetone which produced it (4×58), enable the formation of this substance to be used as a ready means of detecting and estimating traces of acetone.

The mercuric reagent required is prepared by dissolving 5 grammes of mercuric oxide in a hot mixture of 20 c.c. of sulphuric acid and 100 c.c. of water.

For the detection of acetone in aqueous solution, which should not contain more than 10 grammes per litre, 2 c.c. of the solution are mixed with 2 c.c. of the reagent and the tube immersed in a boiling-water bath, a turbidity or precipitate being formed within ten minutes. The lowest limit of the reaction is reached with a solution containing 2 centigrammes per litre, but the smallest traces can be detected by distilling the acetone solution and testing the first tenth of the distillate.

When the acetone is dissolved in methylic alcohol, 2 c.c. of the solution are mixed with 2 c.c. of water, and 4 c.c. of the mercuric reagent added, the dilution being necessary to prevent the precipitation of the mercuric sulphate on boiling the liquid. It is possible to detect 3 centigrammes per litre after the tube has been left for ten minutes in the boiling water.

In the case of solutions in ordinary alcohol, it is necessary to dilute so that the liquid to be tested does not contain more than 2 per cent of alcohol, since otherwise mercurous sulphate is precipitated on boiling. The limit of sensibility is thus less than with aqueous solutions, and may be taken as 1 gramme per litre.

In the quantitative determination of acetone by this method, 25 c.c. of the mercuric reagent are mixed with 25 c.c. of the acetone solution, care being taken that the amount of acetone present does not exceed 50 milligrammes and that the percentage of alcohol in methylic alcohol solutions is not more than 10 or that of ordinary alcohol solutions more than 1 per cent. The flask, with its stopper tied down, is immersed in boiling water for ten minutes. After cooling, the precipitate is collected on a weighed filter, washed with from 75 to 100 c.c. of cold water, dried at 100° C., and weighed. The weight of the precipitate, multiplied by 0·06 (the coefficient experimentally determined), gives the weight of acetone in the 25 c.c. of the solution examined.

The determination may also be made more rapidly by the author's volumetric method of estimating mercury (ANALYST, xxi., 303), the amount of that metal being determined after the precipitation, the amount precipitated being obtained from the difference between the quantity found and that originally present, and the acetone calculated from the result.

The liquid containing the precipitated acetone is diluted to 100 c.c. and filtered. Twenty c.c. of the filtrate are mixed with 15 c.c. of ammonium hydroxide, 50 to 60 c.c. of water, and 10 c.c. of a solution of potassium cyanide, equivalent to a

decinormal solution of silver nitrate. After the addition of a few drops of a solution of potassium iodide (1 : 5) the liquid is titrated with decinormal silver nitrate.

As it is necessary that the solution to be examined should not contain more than a certain amount of acetone, a preliminary approximate estimation of the quantity present may be made by observing the time required for a precipitate or turbidity to form when the mixed liquids are heated in boiling water. Two c.c. of the mercuric reagent and 2 c.c. of the acetone solution, diluted with 9 volumes of water, are mixed in a test-tube 16 to 18 cm. long and 18 mm. in diameter, which is kept in boiling water.

The time required for the appearance of turbidity in the liquid is shown in the following table :

Time.	Acetone Grammes per Litre.	Time.	Acetone Grammes per Litre.
60 seconds ...	10 grammes.	2½ minutes ...	0·20 grammes.
75 ,, ...	5 ,,	3 ,, ...	0·10 ,,
90 ,, ...	2 ,,	4 to 5 minutes ...	0·05 ,,
100 to 105 seconds	1 ,,	10 minutes ...	0·02 ,,
2 minutes ...	0·5 ,,		

C. A. M.

The Purity of Ether used as a Fat Solvent. T. Methner. (*Chem. Zeit.*, 1899, xxiii., 37.)—The *Verband landwirthschaftlicher Versuchsstationen* stipulates that the ether employed to estimate the proportion of fat in food-stuffs shall be free from alcohol and water. Even the purest ethers of commerce contain about 0·5 per cent. of alcohol; and this can only be removed entirely by repeated agitation with fresh clean sodium extended over several days. The author has prepared such ether, and has investigated the yields of fat from various materials in comparison with the same solvent mixed with known quantities (1 to 10 per cent.) of alcohol; also using ordinary ethers of the specific gravities 0·720 and 0·722. His results go to prove that extreme freedom from alcohol is not required; the fat increases slightly as the ether becomes more dilute; but in no case, save where the solvent was damp, did the error exceed 0·25 per cent. calculated on the sample, or 9 per cent. calculated on the fat itself, even when 10 per cent. of alcohol was present in the ether. Considering that food-stuffs do not contain a very large proportion of fat, and that they are not specially rich in substances soluble in alcohol, he holds that commercial ether (specific gravity, 0·720) suffices for all industrial analyses, provided it be thoroughly dried by means of powdered quicklime. F. H. L.

A Comparison of the Methods of Iodine Absorption in the Analysis of Fats. J. J. A. Wijs. (*Chem. Rev. Fett. u. Harz. Ind.*, 1899, vi., 5-11.)—In a recent communication (ANALYST, xxiii., 238) the author showed that in the Hübl process the main reaction is an addition of hypoiodous acid to the fat. He has now made a comparative examination of Hübl's method, of Waller's method (ANALYST, xx., 280). and of his own recently described iodine chloride method (ANALYST, xxiii., 240).

He finds that in the Hübl process the absorption of iodine takes place with great rapidity in the first few moments, and but slowly afterwards. In addition to the length of time, the stage at which the blank determination is titrated has a

considerable influence on the result, as is shown in the following table of iodine values obtained with a freshly prepared solution:

Time of Absorption.	Earthnut Oil. Blank Determination.		Linseed Oil. Blank Determination.	
	Before Absorption.	After Absorption.	Before Absorption.	After Absorption.
2 hours ...	87·02	—	173·74	—
7 ,, ...	88·23	85·38	177·65	170·39
24 ,, ...	90·21	82·32	181·89	163·16

In the author's opinion the results obtained by titrating the blank before the absorption are probably the more correct. For the decrease in the amount of thiosulphate required by Hübl's solution on keeping depends on the oxidation of the alcohol by the hypoiodous acid, and thus on the concentration of that acid. When a fat is present this concentration is somewhat reduced by the addition of part of the hypoiodous acid to the fat, but chiefly by the splitting off of acid (HCl) from the addition compound. Practically the whole of the changes brought about by these reactions occur in the first few moments of the absorption, and thus the oxidation of alcohol by hypoiodous acid in the presence of the fat is very much less than in the blank determination.

With regard to the time allowed for the absorption, the author prefers the iodine value obtained after seven hours, since he finds that the rapidity with which the iodine disappears (as shown by titration) reaches its lowest limit after that time, and then commences to rise again.

With a Hübl solution, five days old, it was found that the absorption occurred more rapidly during the first few moments than was the case with the fresh solution, but that the rapidity of the disappearance of iodine (*i.e.*, I + ICl + HIO) did not reach its lowest limit until after twenty-four hours. The iodine values obtained for the same linseed oil as before with this solution were:

Time of Absorption.	Blank Determination.	
	Before Absorption.	After Absorption.
2 hours	163·82	—
7 ,, ...	172·20	169·00
24 ,, ...	177·60	167·03

In this case the author regards the value 177·60 as the most probable.

Similar determinations were made with Waller's solution. Here it was found that the rapidity of disappearance of iodine (I + ICl + HIO) was considerably less, reaching its lowest point in the case of earthnut oil after twenty-four hours, and with linseed oil only after forty-eight hours. The iodine values thus obtained with the same oils were:

Time of Absorption.	Earthnut Oil. Blank Determination.		Linseed Oil. Blank Determination.	
	Before Absorption.	After Absorption.	Before Absorption.	After Absorption.
24 hours ...	86·98	86·85	170·67	170·11
48 ,, ...	—	—	171·45	170·37

With a strongly acid solution, like that of Waller, the splitting off of hydrochloric acid from the addition compound does not take place, or at most does so to

a trifling extent. Hence the concentration of the hypoiodous acid in the solution remains about the same throughout the absorption, and the author therefore considers the iodine values obtained when the blank is titrated after the absorption as probably the more correct, although the difference is small. As the results obtained by Waller's method are usually somewhat lower than the Hübl value, it is possible that the absorption is not quite complete, even after the twenty-four and forty-eight hours given in the above experiments. The statement frequently made, that the values given by the Waller solution are higher than those of the Hübl process, is to be attributed to the blank determination having been titrated at the end of the absorption.

With the author's solution of iodine chloride in glacial acetic acid (ANALYST, xxiii., 240) the experiments showed that the absorption was complete after fifteen minutes with earthnut oil and after an hour with linseed oil, the further absorption in each case being practically nil.

The following table gives the amount of iodine absorbed by the oils in one minute and its percentage of the actual iodine value in the case of the different solutions :

	Earthnut Oil.		Linseed Oil.	
	I. Absorbed after 1 Minute.	Per cent. of Iodine Value.	I. Absorbed in 1 Minute.	Per cent. of Iodine Value.
Iodine chloride in acetic acid	86·91	98·9	174·07	98·1
Hübl solution, 16 hours old	82·54	93·9	152·46	85·9
,, ,, 5 days old ...	—	—	128·90	72·6
Waller's solution	73·52	83·6	102·64	57·8

The different final values obtained in the experiments described above are thus summarized :

Solution.	Earthnut Oil.			Linseed Oil.		
	Minimum.	Maximum.	Most Probable Value.	Minimum.	Maximum.	Most Probable Value.
Hübl solution, 16 hours old	82·32	90·21	88·23	163·16	181·89	177·65
Hübl solution, 5 days old ...	—	—	—	163·82	177·60	177·60
Waller's solution	86·85	86·98	86·85	170·11	171·45	170·35
Iodine chloride in glacial acetic acid	—	—	87·93	—	—	177·37

In order to determine to what extent the iodine value serves as a measure of the compounds with two unsaturated bonds which occur in fats, the iodine values of a number of unsaturated fatty acids were determined by means of the iodine chloride solution with the following results :

	Melting-point, °C.	Acid Value.		Iodine Value.	
		Calculated.	Found.	Calculated.	Found.
Erucic acid ...	32	165·7	165·2	75·15	74·9
Brassidic acid ...	60	165·7	165·0	75·15	75·0
Elaidic acid ...	44	198·6	198·4	90·07	90·0
Oleic acid* ...	—	198·6	184·3	90·07	87·6
Undecylic acid...	—	304·3	292·9	136·6	133·1

C. A. M.

* Bought as free from linolic acid.

The Heat of Bromination Test for Oils. A. H. Gill and J. Hatch. (*Journ. Amer. Chem. Soc.*, 1899, xxi., 27-29.)—In order to render the results obtained with different calorimeters comparable, the authors propose to refer the rises in temperature to a standard, as in the Maumené test. They find that sublimed camphor can be obtained in a sufficient state of purity to serve as the standard material. The rise of temperature on brominating the camphor is determined in the apparatus and by the method chosen, and the rises in temperature of the various oils are divided by this number, thus giving a "specific temperature reaction."

The following results were thus obtained by a method which was substantially the same as the modification proposed by Wiley (ANALYST, xxi., 210). The factor 17·18 was obtained by dividing several of the Hübl values by this specific temperature.

Oil.	Specific Temperature Reaction.	Iodine Value.	
		Calculated.	Determined (Hübl).
Neat's-foot	3·286	56·5	59·1
Tallow	3·348	57·4	57·2
Prime lard	3·715	63·8	63·8
Sperm	4·191	72·1	73·2
No. 1 lard	4·096	70·3	73·9
Olive	4·762	81·8	82·0
Cotton-seed	5·667	97·3	103·0
Corn	6·381	109·5	107·8
Cod	8·002	137·4	135·0
Linseed	9·049	155·6	160·0

C. A. M.

The Analytical Constants of American Linseed Oil. A. H. Gill and A. C. Lamb. (*Journ. Amer. Chem. Soc.*, 1899, xxi., 29, 30.)—The following results were obtained by the authors with samples of linseed oil representative of all the principal brands in the market. In applying the Maumené test the oil was mixed with a heavy petroleum and allowed to stand for an hour or two before adding the sulphuric acid, a correction being made for the addition. All the results are stated to be the mean of two closely agreeing determinations.

Brand.	Specific Gravity at 15° C.	Valenta Test, °C.	Maumené Test, °C.	Iodine absorbed in 4 hours. Per cent.	Iodine absorbed in 18 hours. Per cent.	Drying Test. Hours required.
Western raw... ...	0·933	79	97	174·7	180	72
,, ,, ...	0·932	70	90	169·7	180	72
,, ,, special	0·934	73	105	178·0	178	72
Old Calcutta... ...	0·931	71·5	106	167·5	178	72
Eastern oil	0·931	73	105	168·0	168	72
Western boiled ...	0·936	74	100	178·8	178·8	18
Eastern ,, ...	0·938	59·5	101	169·5	171	18
Acid bleached ...	0·934	52·5	103	160·0	160	84
Bleached without acid	0·932	60	105	162·0	162	84
Menhaden oil ...	0·934	73·5	135	157·0	181	84
Usual constants ...	0·931—0·937	57—74	103—126	—	170—188	
Average	0·934	—	111	—	176	

It is pointed out that these results differ from the usually accepted values in the Maumené figure being about 10 per cent. higher and the iodine value about 4 per cent. lower. Menhaden oil was examined because it is frequently used to adulterate linseed oil. C. A. M.

The Detection and Estimation of Earthnut Oil in other Oils. J. Bellier. (*Ann. de Chim. anal.*, 1899, iv., 4-9.)—One c.c. of the oil under examination is saponified in a tube with 5 c.c. of an alcoholic solution containing about 85 grammes of potassium hydroxide per litre. 1·5 c.c. of dilute acetic acid, exactly neutralizing the 5 c.c. of the alkali, is then added, and the whole well shaken. The solution of potassium acetate and fatty acids in 70 per cent. alcohol is now cooled rapidly by shaking the tube in water having a temperature below 20° C. Owing to the presence of the potassium acetate the arachidic acid and other solid fatty acids are precipitated in a very short time. When the precipitate ceases to increase, 50 c.c. of 70 per cent. alcohol containing 1 per cent. by volume of hydrochloric acid are added, the tube shaken and placed in water at 17° to 19° C. When the oil contains more than 10 per cent. of earthnut oil, the alcohol leaves a more or less abundant precipitate of arachidic acid. If the quantity is less than 10 per cent., the liquid in the tube remains clear, or nearly so, at first, but after being left for thirty minutes in the water a cloudiness, which masks the bottom of the tube, can be observed. With pure oils, the liquid remains perfectly transparent throughout the whole time.

In this test, the majority of olive oils give a solution which remains absolutely clear even below 16° C. But with certain rare specimens of Tunis olive oil which contain a large amount of solid fatty acids, with cotton-seed oil, and with sesame oil, the liquid may be turbid after the addition of the 70 per cent. alcohol. To distinguish these oils from earthnut oil the temperature is slowly raised until the liquid just becomes clear, and the tube is then placed in water at 17° to 19° C. After thirty minutes, olive, cotton-seed, or sesame oils remain transparent, while in the case of earthnut oil the turbidity is reproduced. The author states that no mistake is possible with an oil containing more than 5 or 6 per cent. of earthnut oil.

For the quantitative determination of earthnut oil, 5 grammes of the oil are saponified with 25 c.c. of the alcoholic potassium hydroxide solution and a volume of dilute acetic acid, exactly neutralizing the alkali taken, is added. The liquid is then rapidly cooled by immersing the vessel in water. After an hour the precipitate is collected on a filter, washed with 70 per cent. alcohol containing 1 per cent. of hydrochloric acid, the temperature being maintained between 15° and 20° C., and the washing continued until the filtrate does not become perceptibly turbid on the addition of water.

The precipitate, which contains other acids besides arachidic acid, is dissolved off the filter with 25 to 50 c.c. of boiling alcohol (92 to 93 per cent.). From 8 to 16 c.c. of water are added to the solution in order to reduce the strength of the alcohol to about 70 per cent., and the liquid is then cooled for an hour in water at a temperature below 20° C. The arachidic acid which precipitates is collected on a dried and weighed filter, washed with 70 per cent. alcohol (free from hydrochloric acid) until

the filtrate remains clear on the addition of water, dried at 100° C. and weighed. The melting-point of the acid thus obtained is usually about 72° C.

From the weight of the acid obtained from pure Bordeaux earthnut oil (0·21 gramme), the coefficient of the oil is $\frac{100}{0{\cdot}21 \times 20} = 23{\cdot}81$. This figure is higher than Renard's coefficient (22). The author considers that this method is capable of rapidly estimating earthnut oil when present in greater proportion than 2 per cent. Of all the oils (other than earthnut oil) examined, only one (Tunis olive oil) gave a precipitate corresponding to about 1·5 per cent. of earthnut oil, as is shown in the following table:

Oil.	Fatty Acid. Per cent. Gramme.	Corresponding to Earthnut oil. Per cent.	Oil.	Fatty Acid. Per cent. Gramme.	Corresponding to Earthnut oil. Per cent.
Bordeaux Earthnut oil	4·20	100·00	Olive oil, Tunis (Sousse)	0·020	0·48
Marseilles ,, ,,	4·17	99·28	,, ,, ,, (Sfax)	0·010	0·24
Olive oil +5% Earthnut oil	0·21	5·00		0·060	1·44
,, ,, +10% ,, ,,	0·42	10·00	African	0·010	0·24
,, ,, +20% ,, ,,	0·84	20·00		0·040	0·95
Olive oil (Nice)	0·030	0·71	Cotton-seed oil (purified)	0·030	0·72
,, ,, ,,	0·030	0·71	Sesame oil (Jaffa)	0·020	0·48
,, ,, ,, (superfine)	0·040	0·95			

The following specimens of olive oil gave only a slight turbidity: Calabrian, Nice superfine, Tunis (Sousse), St. Denis, and Algerian. With olive oils of the following origin the liquid remained perfectly clear: Var olive oil (recent), Var (old), Corsican, Spanish, Italian, Italian (Bari), Nice (fine and superfine) and a sample of unknown origin. C. A. M.

A Method of Separating Citronellal and Citral. **J. Flateau and H. Labbé.** (*Bull. Soc. Chim.*, 1898, xix., 1012-1013.)—The aldehydes citral or geranial ($C_{10}H_{16}O$) and citronellal ($C_{10}H_{18}O$) are found together in the different essential oils of andropogon and in oil of lemon-grass. They can be prepared by the oxidation of their corresponding alcohols, but the yield is small, and in manufacturing processes they are invariably obtained from the essential oils in the form of their bisulphite compounds, and freed more or less completely from other substances by pressure.

The only method of differentiating the two aldehydes hitherto known is that of Doebner, which consists in converting them into the respective citryl- and citronnellyl-β-naphtho-cinchonic acids, which have different melting points, but according to the authors this is only applicable to qualitative work, and cannot be used as a practical means of separation. They have found that on treating an aqueous solution of the bisulphite compounds (prepared by treating the aldehydes for two or three hours with a solution of sodium bisulphite containing a third of its volume of ether, while the temperature is kept low) with a solution of barium chloride, the citryl sulphite

remains in solution, while the citronellyl sulphite is precipitated quantitatively as a barium salt with the composition $C_{20}H_{38}O_8S_2Ba$.

This salt is insoluble in hot alcohol, ether, benzene, and petroleum spirit. It does not dissolve in cold water, but is partially decomposed in boiling water. The citronellal can be recovered by saponifying the salt with a 10 per cent. alcoholic solution of potassium hydroxide, removing the insoluble barium salt by filtration, passing a current of carbon dioxide through the filtrate to remove the excess of alkali, filtering again, precipitating the aldehyde by water, washing it, extracting it with ether, and purifying it by fractional distillation.

By this process the authors found from 5 to 6 per cent. of citronellal in various oils of lemon-grass. The whole of the citral (which is employed commercially as the crude material in the manufacture of ionone) could be recovered from the filtrate from the barium precipitation. C. A. M.

The Characteristics of Essence of Neroli and Essence of Petit-grain. E. Charabot and L. Pillet. (*Bull. Soc. Chim.*, 1898, xix., 853-857.)—The essential oil of petit-grain contains limonene, $C_{10}H_{16}$; linalol, $C_{10}H_{17}OH$ (in the free state and as an acetic ester), a terpene, $C_{15}H_{24}$; and, according to Passy (*Bull. Soc. Chim.*, xvii., 519), geraniol, geranyl acetate, and other oxygenated substances.

According to Tiemann and Semmler (*Berichte*, xxvi., 271) essence of neroli contains about 20 per cent. of limonene, 30 per cent. of lævo-rotatory linalol, and 40 per cent. of linalol acetate. The saponification value given by Schimmel and Co. is 38, but the authors point out that this does not agree with the percentage of ester found by Tiemann and Semmler.

As petit-grain is the oil most frequently employed for the adulteration of oil of neroli, the authors have examined a number of authentic samples which they had themselves distilled. They find that the specific gravity of neroli varies from 0·8720 to 0·8760 at 15° C., and that of petit-grain from 0·8910 to 0·8940.

Other characteristics of the two oils are shown in the subjoined table:

Oil of Neroli.	Rotatory power at 15° C.	Refractive index n_D	Solubility in 80 per cent. alcohol at 20° C.	Essence of Petit-grain.	Rotatory power at 15° C.	Solubility in 80 per cent. alcohol.
	+3·12	1·474 at 18°	2 in 3·1	1	−5·54	2 in 2
2	+3·57	1·475 ,, 18°	2 ,, 2·9	2	−5·12	2 ,, 2·0
3	+2·15	1·473 ,, 20°	2 ,, 2·6	3	−6·15	2 ,, 2·1
4	+3·18	1·473 ,, 20°	2 ,, 2·9	4	−5·57	2 ,, 2·2
5	+4·03	1·473 ,, 20·5°	2 ,, 2·9	5	−5·33	2 ,, 2·1
6	+3·42	1·473 ,, 20°	2 ,, 3·1	6	−4·45	2 ,, 2·1
7	+1·42	1·470 ,, 21°	2 ,, 2·6	7	−6·00	2 ,, 2·1
8	+3·54	1·472 ,, 20°	2 ,, 2·7	8	−5·45	2 ,, 2·2
9	+4·06	1·474 ,, 19·5°	insoluble			

The rotatory power which, contrary to what might be expected from Tiemann and Semmler's researches, is dextro-rotatory, can thus afford a valuable indication of the purity of neroli oil, the dextro-rotation being increased by an addition of oil of

lemon, orange, or bergamot, and reduced by essence of petit-grain. The solubility in alcohol increases with the age of the oil. The insoluble sample (9) was a mixture of different specimens distilled in 1897. A further difference between the two oils is that neroli contains a large proportion of a dextro-rotatory fraction distilling at about 179° C., whereas in the first distillation of petit-grain no fraction is obtained near that temperature.

For a determination of the esters, from 2 to 3 grammes of the oils were heated on the water-bath for thirty minutes with semi-normal alcoholic potassium hydroxide, the residual alkali titrated, and the amount corresponding to the esters expressed as linalol acetate, $C_{10}H_{17}OCO.CH_3$. The following results were thus obtained:

PERCENTAGE OF ESTERS AS LINALOL ACETATE.

No. of sample	1.	2.	3.	4.	5.	6.	7.	8.	9.
Essence of neroli	14·4	10·1	18·0	16·8	13·4	15·1	16·7	12·1	14·5
Essence of petit-grain	54·9	55·9	60·7	59·2	69·6	51·5	61·1	63·7	—

Oil of bergamot contains about 38 per cent. of esters, and an addition of it to neroli would also be shown by the increase in the quantity thus determined.

Since both the oils lost their odour on saponification and acquired that of linalol, the authors consider that their characteristic perfumes are due to the different natures of the esters, the alcoholic constituents of each being apparently identical.

C. A. M.

The Composition of Oil of Thyme. H. Labbé. (*Bull. Soc. Chim.*, 1898, xix., 1009-1011.)—On treating 500 grammes of essential oil of thyme with aqueous solution of potassium hydroxide, the author obtained 160 grammes of thymol. The substances insoluble in the above solution were washed, dried, and fractionally distilled, the following fractions being obtained: Between 155° and 158° C., about 83 grammes; 165°-169°, about 67 grammes; 174°-177° about 70 grammes; 180°-184°, about 30 grammes; 195°-200°, about 15 grammes; 200°-215°, about 28 grammes.

The portion distilling between 155° and 158° was a hydrocarbon, which from the melting-point of its nitroso-chloride (106·5°) was proved to be distinct from pinene, the presence of which was noted by Schimmel and Co. in thyme oil.

The second fraction (165°-169°) had the same boiling-point as menthene, with which substance it was identified by the melting-point of its nitroso-chloride (113°-113·5°), and by the fact that on oxidation with permanganate it yielded cymene.

The fraction which distilled over between 174° and 177° had the characteristic odour of cymene, and like that substance yielded terephthalic acid on oxidation. The fourth fraction (180°-184° C.) also had the odour of cymene, and yielded the same oxidation products. Hence, the author considered it probable that the third and fourth fractions consisted of a mixture of different cymenes boiling at from 174°-184° C.

The next fraction (195°-200°) was identified as linalol by its odour, and by being converted into geraniol on heating in a sealed tube at 150° C. with an equal volume of acetic anhydride.

In the fraction boiling between 200° and 215° C. borneol was identified by Schimmel and Co's. method of oxidizing it to camphor by means of bichromate. The nature of the residue was not determined.

From the results of his determinations the author gives the following as the percentage composition of oil of thyme: Thymol, 30; hydrocarbon (156°-158°), 17; menthene, 15; cymene, 21; linalol, 5; borneol, 8; residue, 4.

He points out that the valuation of the oil should not, as is customary, be based solely on the proportion of phenols which it contains, since the other compounds, such as linalol and borneol, have also pronounced odours, which contribute to and modify the perfume of the essential oil. C. A. M.

On the Estimation of Glucose. Maquenne. (*Bull. Soc. Chim.*, 1898, xix. 926.)—The author simplifies Lehmann's method of determining glucose by titrating the unreduced copper, and by having a large excess of sulphuric acid in the liquid which obviates the filtration. Ten c.c. of Fehling's solution (Pasteur's formula) are mixed with a quantity of the glucose solution containing less than 50 milligrammes, and water added so that the total volume of the liquid is exactly 30 c.c. The whole is then rapidly heated, boiled for two minutes, cooled, and after the addition of 20 c.c. of dilute sulphuric acid (1 : 1) and 10 c.c. of a 10 per cent. solution of potassium iodide, titrated with 2 per cent. thiosulphate, using starch as indicator.

The difference between the number of c.c. required by the original Fehling's solution and by the liquid after titration gives the amount corresponding to the glucose.

The following table gives the volume of the thiosulphate solution corresponding to known quantities of saccharose inverted by the ordinary method.

Sugar, milligrammes.		Thiosulphate, c.c.	Sugar, milligrammes.		Thiosulphate, c.c.
2·5	...	0·90	30	...	10·95
5	...	1·80	35	...	12·70
10	...	3·75	40	...	14·45
15	...	5·65	45	...	16·15
20	...	7·45	50	...	17·80
25	...	9·20			

C. A. M.

Test for Albumin in Urine. W. C. Alpers. (*Amer. Journ. Pharm.*, 1898, lxx. 449-451.)—The test for albumin consists in acidifying the urine with hydrochloric acid, and adding an equal volume of a 1 per cent. solution of mercury succinimide. In the presence of as little as 1 part of albumin in 150,000, a white cloudiness is obtained. C. A. M.

The Influence of Food Preservatives on Digestive Enzymes. H. Leffmann. (*Journ. Franklin Instit.*, 1899, cxlvii., 97-108.)—The enzymes used in the author's experiments, which had reference principally to the digestion of starch, were: Malt diastase (Merck); pancreatic extract (Parke, Davis and Co.); peptenzyme, a preparation containing all the digestive ferments of the alimentary tract (Read and Carnick);

and papaïn, the enzyme of papaw sold under the name of "caroid." In each experiment 50 c.c. of a freshly-prepared solution of arrowroot-starch (10 grammes per litre) were used.

The following results were obtained in a series of experiments with taka-diastase 11 milligrammes of the enzyme and 50 milligrammes of the preservatives being used in each case, and the solutions diluted to 150 c.c. after the digestion:

1. Salicylic Acid: Showed starch reaction strongly, and only traces of sugar; 133 c.c. of the solution did not reduce 10 c.c. of copper solution (equivalent to 0·046 gramme of dextrose).

2. Sodium Benzoate: No starch present; much sugar formed.

3. Saccharin: Much starch present; traces of sugar.

4. Boric Acid: No starch present; much sugar formed.

With pancreatic extract solution the results were:

	Milligrammes.	Pancreatic Solution, c.c.	
Salicylic acid... ...	50	1	No sugar formed.
Sodium benzoate ...	50	1	Much sugar formed.
Saccharin	50	1	No sugar formed.
Boric acid	50	1	Much sugar formed.
Salicylic acid... ...	50	3	No sugar formed.
Sodium benzoate ...	50	3	Much sugar formed.
Saccharin	50	3	Trace of sugar formed.
Boric acid	50	3	Much sugar formed.

Saccha[illegible] in small quantities (10 milligrammes) interfered with the digestion of starch b[illegible] taka-diastase (10 milligrammes), by papaïn (20 milligrammes), and to a lesser extent by peptenzyme. Formalin (3 c.c.) did not affect taka-diastase (11 milligrammes), but 1 c.c. had an injurious effect on pancreatic extract (1 c.c.).

From the results of these and similar experiments which are given in tabular form, the author has arrived at the following general conclusions: Beta-naphthol is injurious to malt diastase, but does not seriously affect the starch-converting capacity of the taka-diastase or pancreatic extract. Boric acid, borax and boroglyceride, interfere but little with either starch or proteid digestion. Salicylic acid interferes with the action of most of the enzymes, especially those that convert starch, but does not seriously affect proteid digestion. Sodium benzoate has no appreciably injurious influence on any of the enzymes. Sodium fluoride interferes but little with the digestion of starch, but sodium silicofluoride has a considerable influence on pancreatic extract.

In the author's opinion, if the use of any preservative is to be permitted in food, boric acid and sodium benzoate are the least objectionable, since they appear to have less tendency to disturb the digestive functions than the other preservatives commonly employed. C. A. M.

Estimation of Humus in Soil. C. Aschmann and H. Faber. (*Chem. Zeit.*, 1899, xxiii., 61.)—This process depends on dissolving the humic acid in alkali, and titrating the liquid with permanganate. Solutions required: Sodium hydroxide

50 grammes per litre; permanganate, 0·32 gramme of the potassium salt per litre; oxalic acid, 0·63 gramme per litre; 1 : 5 sulphuric acid. To standardize the reagent, the strength of the oxalic acid must first be ascertained in terms of the permanganate. 0·125 gramme of pure humic acid (Merck) is dissolved in 500 c.c. of water with the aid of sodium hydroxide. To 5 c.c. of the solution and 100 c.c. of water permanganate is added until the colour remains constant; after five minutes 10 c.c. of oxalic acid are added, and the end-point of the reaction is determined in the usual manner by titrating the excess with permanganate. In carrying out the actual analysis, 25 grammes of air-dried, finely sieved earth are heated for an hour on the water-bath with 100 c.c. of the sodium hydroxide solution. The solution is removed by decantation and the insoluble matter treated similarly several times. The liquid is finally diluted to 510 c.c. (allowing 10 c.c. for the deposit), and after standing some days to clarify (filtration is to be avoided), is ready for titration, 5 c.c. being manipulated exactly as above. In order to obtain accurate results, the proportion of humic acid present in the extract should not greatly exceed that employed during the standardization of the permanganate, a point which can be settled by comparison of their respective colours, and adjustment of the volume of the solution obtained from the soil.

As examples of the amount of humic acid in various soils, the following may be quoted:

	Per cent.
Good garden mould	1·87
Highly manured hotbed	5·5—11·5
Forest earth, upper layer	0·88
,, ,, subsoil	0·3
Soil from tan-pit, upper layer	1·25
,, ,, under ground	0·44
Field earth, extremes	0·05—1·7
,, ,, average	0·5—0·7

F. H. L.

Combustion of Mixed Gases containing Carbon Monoxide, Methane, and Hydrogen. L. M. Dennis and C. G. Hopkins. (*Zeits. anorg. Chem.*, 1899, xix., 179.)—The authors remark that there are objections to all processes for the analysis of gaseous mixtures which depend on absorption of hydrogen with palladium or of carbon monoxide with cuprous chloride; and they fall back on the old explosion methods, so modified by the aid of suitable apparatus as to avoid liability to excessive pressure, which may cause the formation of oxides of nitrogen, if not actual danger to the operator. The apparatus which they have adopted resembles that of Winkler, and is shown in the accompanying illustrations. The rubber cork *a* (Fig. 1) carries a glass tube *b* open at both ends, in which a copper wire *c*, 3 mm. thick, is held air-tight by a rubber ring. The second copper wire, 1·5 mm. in diameter, is pushed through the cork and twisted round the glass tube till it reaches the top of *c*; and a platinum spiral *d*, of twenty or thirty coils, 2 mm. in diameter, made of wire 0·25 mm. thick, the ends of which are wrapped round the copper conductors, forms the source of heat. The general arrangement of the apparatus is

explained by Fig. 2: *o*, *o* are clips; *k* is a screw clamp. The measuring vessel is an ordinary Hempel burette fitted with a water-jacket. *F* is a means of varying the resistance, so that *d* may be kept at the desired temperature. The sealing liquid is mercury which bears a drop of water on its surface to insure perfect saturation. The volume of gas operated on is selected so that neither it, nor the necessary oxygen, nor the residue after explosion, shall exceed 100 c.c. The pipette is filled with mercury by opening *g* and raising the reservoir, then closing *g* and applying a vacuum pump to *t* till the air in *b* is extracted. A measured quantity of the sample is introduced, and a known excess of oxygen brought into the burette, which are both kept at about atmospheric pressure. *k* is closed, the burette-reservoir is raised to such a position that the mercury shall eventually fill the burette entirely without passing over into the pipette. Electrical connections are then made, the spiral heated to redness, *o*, *o* are opened, and *k* is cautiously unscrewed till the oxygen flows into the pipette at about 10 or 20 c.c. per minute. The exact speed is chosen so that the spiral remains incandescent, and it needs to be increased in inverse proportion to the molecular weight of the material examined. The combustion proceeds quietly without flame or explosion; but if pure hydrogen be under treatment, it is necessary, owing to the excessive contraction, to employ a mixture of oxygen and air, and also to diminish the force of the current as the operation proceeds, lest the platinum melt in the final atmosphere of oxygen and nitrogen.

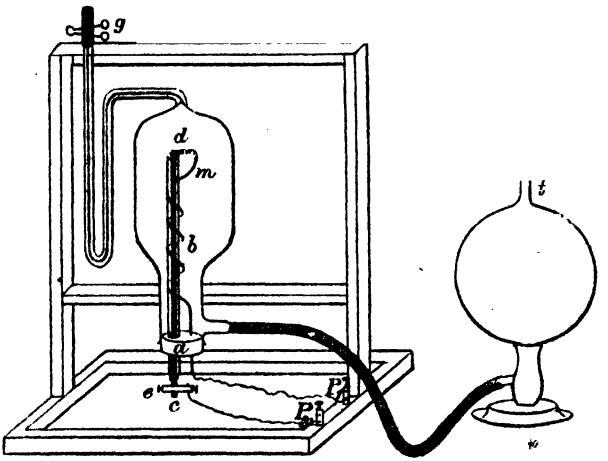


FIGURE 1.

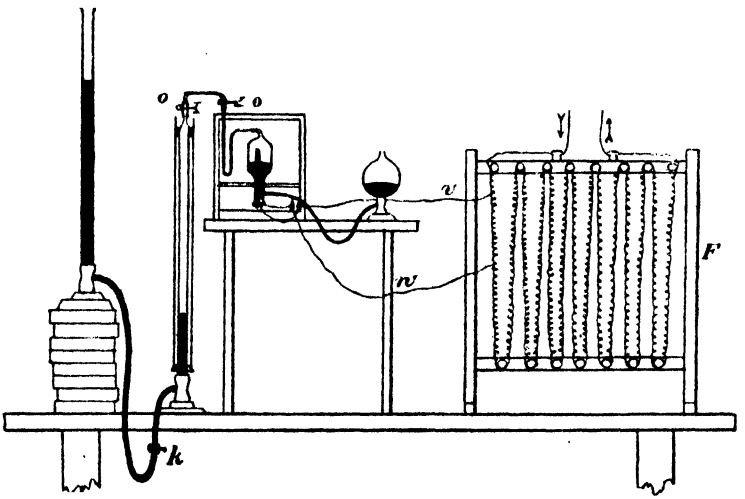


FIGURE 2.

Mixtures of hydrogen, methane, and nitrogen were prepared from ordinary coal-gas by absorbing the heavy hydrocarbons in fuming sulphuric acid, the oxygen in alkaline pyrogallol, and the carbon monoxide by double treatment with cuprous chloride. A known volume (60 to 67 c.c.) of the gas was then burnt with a known amount of oxygen, the residual volume was noted, the carbon dioxide absorbed by potassium hydroxide, and the final residue measured. Five successive tests showed from 56·4 to 56·6 (mean 56·44) per cent. by volume of hydrogen, and 39·9 to 40·0 (mean 39·94) of methane; corresponding experiments with 12 c.c. in a Hempel pipette giving 55·85 of hydrogen and 41·10 methane. Mixtures of hydrogen, carbon monoxide, methane, and nitrogen similarly prepared yielded equally satisfactory results, the greatest variation from the average proportion of hydrogen being 0·37 per cent. when analysing a mixture containing 53·7 per cent.

In the latter case the process is carried out as follows: A measured volume of the gas is burnt with a measured excess of oxygen, the residue is driven over into the burette and measured, the carbon dioxide is removed by potassium hydroxide, and the volume again noted. A known amount of hydrogen is introduced into the pipette, the current is again started, and the remaining mixture of nitrogen and oxygen forced back gradually until all the gas is in the combustion pipette once more. From the amount of contraction which then occurs the excess of oxygen can be calculated, and the difference between this figure and the original quantity of oxygen employed gives the volume of oxygen actually used in the first combustion. By means of these data the composition of the gaseous mixture may be deduced thus: V = the original volume of the sample; K = the contraction during the (first) "explosion"; O = the volume of oxygen required to oxidize V; CO_2 = the volume of carbon dioxide already determined:

1. $H = K - O$.
2. $CO = \frac{4CO_2 + H - 2O}{3}$.
3. $CH_4 = CO_2 - CO$.
4. $N = V - (H + CO + CH_4)$, or,
5. $CO = CO_2 - CH_4$.
6. $CH_4 = \frac{2K - CO_2 - 3H}{3}$.

In the absence of nitrogen, these formulæ may be substituted:

7. $H = V - CO_2$.
8. $CO = \frac{CO_2 + 3V - 2K}{3}$.
9. $CH_4 = \frac{2CO_2 + 2K - 3V}{3}$.

F. H. L.

The Calculation of the Calorific Power of Coal from Analytical Data. D. De Paepe. (*Bull. de l'Ass. belge.*, 1898, xii., 279-302.)—The author has studied the various methods of calculating the calorific power of coal, comparing the results with actual calorimetric determinations. He has obtained good results by Mahler's formula (*Bull. de la Soc. d'encour. pour l'ind.*, 1892, vii., 358):

$$P = 8140C + [34{\cdot}500H - 3000(O+N)],$$

in which P represents the calorific power, and C, H, O and N the respective percentages of carbon, hydrogen, oxygen and nitrogen. This method, however, has the drawback that elementary analysis is too tedious and delicate for ordinary technical analytical work.

E. Goutal (*Rev. de Chim. ind.*, 1896, vii., 65) has proposed the following formula as being more readily applicable:

$$P = 8150C + \frac{AM}{100},$$

in which P represents the calorific power; C, the percentage of fixed carbon (= coke —ash); M, the percentage of volatile matter (= 100—[coke+water]); and A, a co-efficient which varies with the amount of volatile matter: viz., $A = 13{,}000$ for $M = 2$ to 15; $A = 10{,}000$ for $M = 15$ to 30; $A = 9{,}500$ for $M = 30$ to 35; and $A = 9{,}000$ for $M = 35$ to 40.

According to the author's experiments, the results obtained by the use of this formula agree well with those calculated by Mahler's formula, or actually determined, if the following values for A are substituted for those given by Goutal. Where M' is used to represent the amount of volatile matter calculated on the coal supposed to be deprived of its water and ash, $M' = \frac{100M}{(M+C)}$

$A = 14{,}000$ for $M' = 2$ to 12	$A = 9{,}400$ for $M' = 30$ to 35
$= 12{,}000$,, $= 12$ to 17	$= 8{,}000$,, $= 35$ to 38
$= 11{,}000$,, $= 17$ to 24	$= 7{,}900$,, $= 38$ to 40
$= 10{,}200$,, $= 24$ to 30	$= 7{,}600$,, $= 40$ to 50

The accuracy of the calculations with the modified formula is illustrated by a long series of tables, of which the following, giving its application to the analytical results of Noyes, McTaggart and Craver (ANALYST, xxi., 22), may be quoted.

Coal.	Calorific Power calculated by Goutal's modified formula.	Calorific Power determined with Hempel's calorimeter.	Difference.	Percentage Difference.
1. New Pitsburgh, A	6,288	6,175	+113	+1·85
2. New Pitsburgh, B	6,502	6,415	+87	+1·35
3. Lancaster	6,694	6,703	—7	—0·10
4. Brazil	6,721	6,846	—125	—1·90
5. Shelburn	6,492	6,532	—40	—0·60
6. Shop	5,823	5,806	+17	+0·30

C. A. M.

INORGANIC ANALYSIS.

Electrolytic Estimation of Tin in Tin Ores. E. D. Campbell and E. C. Champion. (*Journ. Amer. Chem. Soc.*, 1898, xx., 687-690.)—One gramme of the finely-powdered ore is treated with sodium carbonate and sulphur, in the usual manner. When cold the mass is dissolved in 40 to 50 c.c. of water, and filtered from the insoluble oxides and sulphides. The solution contains nearly all the tin in the form of sodium sulphostannate, and the small quantity left in the residue is fused as before and brought into solution. The united solutions are made weakly acid with hydrochloric acid, and the tin precipitated as tin sulphide. The liquid which contains the stannic sulphide in suspension is then boiled to expel the excess of hydrogen sulphide, and concentrated to about 75 to 80 c.c. Ten c.c. of hydrochloric acid (specific gravity 1·20) are added to the hot solution, and small quantities of sodium peroxide stirred in until the tin sulphide is completely converted into oxide and the solution has become clear. The liquid is boiled two or three minutes, filtered from the sulphur into a large platinum dish, and the residue washed with hot water. Ammonia is added to the solution until a permanent precipitate results, and then 50 c.c. of a 10 per cent. solution of ammonium oxalate, and the clear liquid is electrolyzed over night with a current of N D_{100} = 0·10 A and 4 V. When the tin has been completely precipitated the deposit is washed with water and alcohol and dried at 80 to 90° C., and the dish weighed.

The deposit is finally dissolved off the basin with dilute hydrochloric acid, and the weight of the washed and dried platinum basin determined. C. A. M.

Iodometric Estimation of Gold and Platinum. H. Peterson. (*Zeits. anorg. Chem.*, 1899, xix., 59.)—The De Haen process for the determination of copper (Fresenius, *Quant. Anal.*, i., 335), which depends on the addition of an excess of potassium iodide to a solution of copper free from any substances capable of decomposing the reagent, and titrating the liberated halogen by means of thiosulphate, is equally available in the case of gold and platinum. With the latter, the dark reddish-brown liquid produced when a strong solution of iodide in excess is mixed with the double platinic chloride should be treated with standard thiosulphate (without starch) till the colour changes to a pale lemon yellow; 1 atom of platinum corresponding to 2 molecules of thiosulphate. Gold is similarly treated in the presence of starch; but, as an intermediate reaction takes place between the aurous iodide and part of the reagent yielding the compound $NaAuS_2O_3$, 1 atom of gold corresponds to 3 of thiosulphate. Both methods should be useful where only small amounts of the respective metals have to be estimated; and in many instances cupellation of gold samples may thus be avoided. F. H. L.

Qualitative Separation of the Platinum Metals. F. Mylius and R. Dietz. (*Ber.*, 1898, xxxi., 3191.)—The following process is published as an appendix to an article on the characteristics and purity of the several platinum metals now met

with in commerce; it does not profess to give absolutely complete separations, but is the simplest that can be devised. The amount of material operated upon should contain about one gramme of the platinum metals; and beside gold and mercury, the common metals may be present in the neutral or acid solution as chlorides. Platinum alloys may be run down with lead, which converts iridium and ruthenium into an insoluble form, leaving the others soluble. The solution, mixed with nitric acid, is distilled in a retort and the vapours led into caustic soda. If the liquid becomes yellow, *osmium* may be present, and can be identified by the odour of its tetroxide on addition of hydrochloric acid, or by the production of its brown sulphide when the acid liquor is warmed with thiosulphate. The cold residue from the retort is extracted, three times if necessary, with a little ether. Should this become yellow, *gold* is indicated (chromic acid also gives a colour varying from yellow to blue, and ferric chloride may equally tint the ether), and may be tested for with ferrous sulphate, etc. The residual solution is treated with ammonium acetate, and boiled for several hours with formic acid under an inverted condenser in order to throw down the noble metals. The precipitate is filtered off, washed with acetate solution, dried and ignited in a porcelain boat in a stream of hydrogen. *Mercury* yields the well-known metallic sublimate. The residue is extracted with warm HCl (to remove traces of tin and other heavy metals not reduced by the formic acid), dried, mixed with common salt, heated as before to dull redness in a current of damp chlorine, and the product leached with water. Any insoluble matter must be repeatedly submitted to the same process; should any portion resist the chlorine, it must be further examined for iridium and ruthenium. To the aqueous solution from the ignition in chlorine, saturated ammonium chloride is added as long as a precipitate (B) falls, which should consist of Pt, Ir, and Ru; Rh and Pd remaining theoretically in solution (A). If dark in colour, (B) is dissolved in warm water, treated with hydroxylamine hydrochloride, cooled, and precipitated again with ammonium chloride. The deposit is recrystallized twice, if necessary, and identified as *platinum* under the microscope. The hydroxylamine converts the bichlorides of Ir and Ru into sesquichlorides; their solution is evaporated to dryness, ignited in hydrogen, and fused with nitre and potash in a silver crucible. The melt is attacked with water and separated by decantation into a blue-black residue (C) and a solution (D). (D) is saturated with chlorine, and distilled from a retort as long as the vapours are coloured into weak alcohol containing HCl. A yellowish-brown colour indicates *ruthenium*, which, volatilizing as tetroxide, now exists as chloride. Its presence may be corroborated by rendering the liquid ammoniacal and adding thiosulphate: a strong red-violet colour is produced. (C) is washed, dried, ignited with salt and chlorine, and taken up in water; *iridium* yields a dark-brown solution, from which ammonium chloride precipitates the double salt. (A) is slowly evaporated with excess of ammonia, and the residue crystallized from weak cold ammonia; any insoluble matter, consisting generally of Ru, is removed by filtration. *Rhodium* separates as yellowish crystals of $Rh(NH_3)_5Cl_3$, soluble in hot, strong sulphuric acid with a yellow colour. (It should be noted that ignition with salt and chlorine gives a pink aqueous extract, from which Rh is not thrown down by NH_4Cl in dilute solution, *cf.* A and B above.) The ammoniacal filtrate is acidified with strong HCl

when *palladium* falls as $Pd(NH_3)_2Cl_2$. It is purified by ignition in hydrogen, solution in nitric acid, evaporation, dissolution in water, and precipitation with mercuric cyanide. F. H. L.

Estimation of Manganese by means of Potassium Permanganate. F. W. Daw. (*Chemical News*, vol. lxxix. [2043], p. 25.)—To obviate the sources of error usually present—non-elimination of organic matter, excessive use of zinc oxide, and standardizing the reagent against iron instead of manganese—the author proposes the following method: 0·5 gramme of spiegel or ferro-manganese—together, in the latter case, with 0·4 gramme of pure iron wire—is dissolved in 30 c.c. of hydrochloric acid (the iron being oxidized with a little nitric acid), and 15 c.c. of 50 per cent. sulphuric acid added. After evaporation until a copious disengagement of acid fumes ensues, the sulphates are taken up with water and the solution made up to about 500 c.c. The acid is then partly neutralized with sodium carbonate, and the iron thrown down by a gradual addition of zinc oxide, a large excess being avoided. The whole is boiled up, and, without filtering, titrated with permanganate solution (10 grammes per litre, standardised against ferro-manganese of known composition).

In estimating the manganese in the standard sample, the author precipitates with bromine and ammonia, and weighs as mangano-manganic oxide. Silica and metallic oxides are removed by dissolving in hydrochloric acid, adding ammonium acetate, and passing hydrogen sulphide, the weight of the resulting precipitate, after ignition, being deducted from the result of the first weighing. C. S.

Separation of Cerium from Lanthanum and "Didymium." P. Mengel. (*Zeits. anorg. Chem.*, 1899, xix., 67.)—The author has investigated the reactions of the cerium group of metals singly and in combination, studying their behaviour when ignited in the air, and using specially prepared and purified specimens of cerium salts or artificially made mixtures of cerium oxides with those of lanthanum and "didymium." He finds that of all the oxidizing agents hitherto proposed for the isolation of cerium, sodium peroxide is the most convenient; it is available in all cases, and gives the largest yields. When the various oxalates of this group of metals are strongly ignited together, the whole of the cerium is converted into dioxide. When a mixture of the oxides of lanthanum and didymium is heated to 400° or 500° C., it takes up oxygen and becomes brown in colour; at a white-heat the surplus oxygen is expelled, and the colour changes to gray. But if cerium be present simultaneously, this decolorization does not occur; for a definite brown compound of cerium dioxide and praseodymium peroxide (Pr_4O_7) is produced which is permanent at high temperatures. For this reason processes for the determination of cerium that depend on oxidation followed by ignition are incorrect, and only give approximate results when but little praseodymium occurs in the sample.

F. H. L.

The Separation of Rubidium and Cæsium. A. Lemoine. (*Bull. de l'Ass. belge*, 1898, xii., 344-847.)—A usual method of separating these metals consists in

treating the mixed carbonates with absolute alcohol, which dissolves cæsium carbonate, leaving rubidium carbonate, which is nearly insoluble. Various methods have been proposed for converting the chlorides into carbonates, but the author finds the following the most simple and rapid: The residue of chlorides is warmed for some time on the water-bath, with frequent stirring, with an excess of silver carbonate suspended in water. The liquid is filtered, the precipitate washed with cold water, and the filtrate containing the rubidium and cæsium as carbonates evaporated to dryness and treated with alcohol in the usual manner.

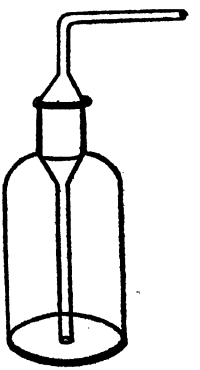
C. A. M.

Copper as a Reagent for Polysulphides in Ammonium Sulphide. T. Chandelon. (*Bull. de l'Ass. belge*, 1898, xii., 277.)—On introducing a strip of bright copper into a solution of ammonium sulphide free from polysulphides, the metal remains bright for several days, whereas if only a minute quantity of a polysulphide be present it becomes black within a few hours. By means of this reaction the author has found that traces of polysulphides are formed when a dilute solution of ammonia is saturated with hydrogen sulphide. He has proved that the blackening of the copper is not due to any excess of hydrogen sulphide in the liquid, a saturated aqueous solution having no action on the metal, at least during the first hours of contact. C. A. M.

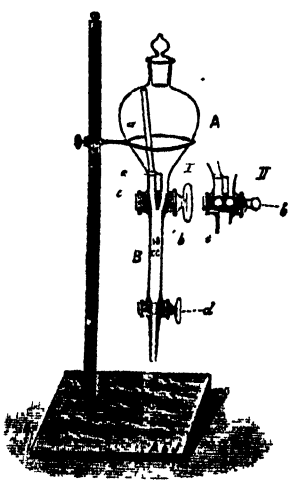
Copper as a Reagent for Tetrathionates in Sodium Thiosulphate. T. Chandelon. (*Bull. de l'Ass. belge*, 1898, xii., 277-279.)—On treating metallic copper with a solution of a tetrathionate there is an immediate abundant deposit of copper sulphide, with the formation (probably) of tri- or penta-thionate, or perhaps a mixture of the two. By means of this reaction it is possible to detect traces of tetrathionate in a solution of thiosulphate. From an 8 per cent. aqueous solution of sodium thiosulphate sold as "chemically pure" the author obtained a very adherent deposit on the copper within twenty-four hours. C. A. M.

APPARATUS.

Apparatus for the Employment of Sulphuretted Hydrogen. C. Graebe. (*Ber.*, 1898, xxxi., 2981.)—Attention is called to the advantages, both on the score of purity of the laboratory atmosphere and economy in raw material, accruing from the adoption of closed vessels for the treatment of liquids with this reagent. A convenient form of flask is shown in the annexed sketch; it is constructed of such dimensions that the solution only fills it about half-way. The supernatant air is driven out before the stopper is fully inserted, and then the gas simply passes into the vessel at the rate it is absorbed; the operation may thus be left over night, or can be hastened by periodical agitation. F. H. L.



Apparatus for Distillation in High Vacua. L. T. C. Schey. (*Chem. Zeit.*, 1899, xxiii., 61.)—This apparatus, illustrated in the accompanying sketch, is designed for distillations *in vacuo* with the aid of a water and mercury pump. As will be seen, it has only one rubber connection. The long-necked flask is charged through a tube funnel, and the opening is then sealed; at the end of the distillation a piece of rubber tubing is slipped over the point, which is broken to allow dry air to enter. The flask is heated in an air-bath, the temperature of which alone is noted. The receiver is cooled with an ice-jacket. When the distillate has been collected, the lower bulb is cut off or removed by fusion. By joining a fresh bulb in the same place, the whole apparatus is ready for use again, and can be employed as long as sufficient neck remains to the distilling flask. It is convenient to use two similar apparatus, one being at work while the other is mended and filled. F. H. L.



An Automatic Reservoir Pipette. H. Göckel (*Zeits. angew. Chem.*, 1899, 10.) The construction of this apparatus is shown by the annexed diagram. The stopcock *b* has two parallel holes, and also a groove *c* which serves for the admission of air when the pipette *B* is being emptied. Care must be taken that no liquid (or water when the pipette is washed) passes into *a* otherwise the pipette works quickly and accurately It is made in various sizes by Alt, Eberhardt, and Jager of Ilmenau. F. H. L.

THE ANALYST.

MAY, 1899.

PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS.

THE monthly meeting of the Society was held on Wednesday evening, April 12, in the Chemical Society's Rooms, Burlington House, the President, Mr. W. W. Fisher, M.A., occupying the chair.

The minutes of the previous meeting were read and confirmed.

Certificates of proposal for election to membership in favour of the following candidates were read for the second time: Julian L. Baker, F.I.C.; Herbert Edward Burgess; Louis Charles Deverell; Julius Lewkowitsch; F. R. O'Shaughnessy; Arthur Marshall; and Edward T. Shelbourn; and in favour of the following candidates for the first time: Edward Halliwell, F.I.C., Analyst to the West Riding of Yorkshire Rivers Board, Wakefield; Arthur John Starey, assistant to Mr. Lawrence Briant, London; and William Lincolne Sutton, F.I.C., Public Analyst for West Suffolk, East Suffolk, and the boroughs of Norwich, Ipswich, and Bury St. Edmunds.

The following were elected members of the Society: Percy T. Adams, Arthur French Angell, John B. Ashworth, Alfred Joseph Bull, Charles Crocker, John S. Ford, C. A. Hackman, Norman Leonard, J. F. Liverseege, Richard Murray, G. E. Scott-Smith, W. P. Skertchly, Harry Metcalfe Smith, Lionel W. Stansell, and W. Northfield Yarrow.

The PRESIDENT (Mr. Fisher) said that the Council had been considering the Sale of Food and Drugs Bill recently introduced in the House of Commons by Mr. Walter H. Long (President of the Board of Agriculture), the Solicitor-General, and Mr. T. W. Russell, and had thought it advisable to make some representation to the President of the Board of Agriculture, and to bring before him in a definite way the views of the Society of Public Analysts. In the forefront of the interview which Mr. Long had accorded to the representatives of the Council was placed the strong feeling which the Council held that there was a necessity for the establishment of what had been termed a Standing Departmental Committee or Court of Reference. The Bill provided that the Board of Agriculture should, after such inquiry as it might deem fit, issue regulations having relation to certain agricultural products. It was represented to Mr. Long, on behalf of the Council, that they would still desire to see a wider and more comprehensive body established, failing which it was suggested that the Local Government Board should have equal powers with the Board of Agriculture to confer with a strong scientific body in reference to other articles of food and to drugs. Mr. Long did not make any distinct promise on this matter, but it

might fairly be said that he received the Council's representation sympathetically. In addition to the provision which the Bill contained, that the Board of Agriculture should decide what proportions of extraneous matters were to be permitted in certain articles of food, the Council asked that the subject of preservatives should be definitely mentioned. A suggestion was made with the object of rendering clause 8, which dealt with the admixture of butter-fat with margarine, more clear and definite in meaning; and also that there should, if possible, be embodied in the Bill definitions of skimmed and separated milk in terms of the percentages of residual fat which they respectively contained. Other suggestions were offered by the Council respecting the effect of the time limit of twenty-eight days in proceedings against the giver of a warranty; the reference of samples to the Commissioners of Inland Revenue; the form of certificates; and the embodiment in the Bill of some definition by which the word "food" might be made to apply to articles which, although not themselves strictly of the nature of food, were used in the preparation or flavouring of food.

The following papers were read: "On a recent Ginger Case," by E. J. Bevan, Bernard Dyer, D.Sc., and Otto Hehner; "The Composition of Milk and Milk Products," "An Attempt to compute mathematically the most Probable Limits for Milk," and a "Note on the Estimation of Ether and Alcohol when mixed with Petroleum Ether and other Solvents," by H. Droop Richmond; "The Estimation of Fat in Milk, using Petroleum Ether as a Solvent," by H. Droop Richmond and C. H. Rosier; and "Notes on Milk Analysis, including the Molybdate Test for Cane Sugar," by L. de Koningh.

Mr. W. P. Skertchly exhibited a new form of distilling flask for use in water analysis.

A METHOD FOR THE QUANTITATIVE SEPARATION OF ISOVALERIC AND ACETIC ACIDS.

By Alfred C. Chapman, F.I.C.

(*Read at the Meeting on November* 2, 1898.)

The quantitative separation of small quantities of isovaleric and acetic acids is not by any means a simple matter. The method of partial neutralization followed by distillation is in this case practically useless, whilst the ingenious method of Duclaux, although affording an approximate determination of the proportions in which the two acids exist, does not affect their separation. When a solution containing the above two acids free from their homologues has been obtained, they may, of course, be estimated by neutralization with baryta, followed by a determination of the percentage of barium in the mixed salts. This indirect method is open to the objection that any failure to obtain the mixed barium salts in a pure and dry condition (by no means an easy matter) will introduce a very appreciable error into both determinations.

Moreover, it is not, strictly speaking, a method for their separation. Having frequently had occasion to estimate the percentages of these two acids when present together in solution, and if possible to obtain them separately for purposes of further

identification, I was led to make experiments with this object in view. At the outset it seemed likely that a difference of solubility of a metallic acetate and of the corresponding valerate in some solvent might offer the best chances of success.

Sodium valerate dissolves fairly readily in a solution containing 99·5 parts of acetone and 0·5 part of water, whilst sodium acetate is practically insoluble in that menstruum. The 99·5 per cent. acetone is, in fact, an excellent solvent from which to obtain pure sodium valerate in a crystalline condition. If the commercially pure salt is treated with a sufficient quantity of the solvent at its boiling-point and filtered, beautiful crystals having a pearly lustre separate from the filtrate on cooling, which can readily be filtered by the aid of a pump, and dried in a vacuum over sulphuric acid. The salt purified in this manner was used for the experiments described below. A determination of sodium showed it to be perfectly pure:

Found, 18·46 per cent. Na. Calculated, 18·54 per cent. Na.

It was in the first place necessary to determine the best conditions for the application of this method, and to ascertain with what degree of accuracy the separation could be effected. Known weights of dry sodium valerate and anhydrous sodium acetate (both powdered) were transferred to a small flask and thoroughly mixed. The requisite quantity of the 99·5 per cent. acetone was then added, and the mixture heated just to its boiling-point for some minutes, a piece of glass tubing passing through a cork in the flask serving to prevent the loss of any appreciable quantity of the acetone. The liquid was then passed through a small asbestos filter into a second small flask, and the residue in the first flask washed with a further quantity of the acetone in two successive portions. The acetone was then distilled off, the residue washed into a platinum dish, evaporated to dryness, treated with sulphuric acid, and weighed as sodium sulphate. The sodium acetate remaining in the first flask and on the funnel was then dissolved in water, and treated in a similar manner. From the weights of sodium sulphate obtained the amount of acetic and valeric acids can of course be easily calculated.

In the following table some of the results obtained are given:

Taken.			Found.		
Sod. Acetate.	Sod. Valerate.	Total.	Sod. Acetate.	Sod. Valerate.	Total.
0·122 gr.	0·109 gr.	0·231 gr.	0·122 gr.	0·106 gr.	0·228 gr.
0·124 gr.	0·117 gr.	0·241 gr.	0·123 gr.	0·118 gr.	0·241 gr.
0·144 gr.	0·093 gr.	0·237 gr.	0·143 gr.	0·090 gr.	0·233 gr.
0·067 gr.	0·104 gr.	0·171 gr.	0·066 gr.	0·103 gr.	0·169 gr.
0·250 gr.	0·250 gr.	0·500 gr.	0·246 gr.	0·254 gr.	0·500 gr.
0·217 gr.	0·238 gr.	0·455 gr.	0·217 gr.	0·235 gr.	0·452 gr.
0·210 gr.	0·145 gr.	0·355 gr.	0·211 gr.	0·142 gr.	0·353 gr.

Potassium valerate is also soluble in 99·5 per cent. acetone, and can be obtained from it in pearly plates. Although potassium acetate is almost insoluble in that liquid, the potassium salts cannot be used for the purpose of separation by this method, as the valerate abstracts water from the solvent, and the acetate is converted into a pasty mass.

Experiments were next made starting with the acids themselves. Aqueous

solutions of the two acids were prepared, and their strengths determined by titration with standard alkali.

The isovaleric acid used had a constant boiling-point of 176°, and yielded a sodium salt containing the theoretical percentage of the metal. Definite volumes of these solutions were mixed and carefully neutralized with a solution of pure caustic soda. The flask containing the solution of the mixed sodium salts was then connected with a condenser and the water distilled off under reduced pressure. In this way the slight loss of valeric acid which usually occurs when solutions of the metallic valerates are evaporated in contact with the air, was avoided, and the rate of evaporation was of course accelerated. The residue was dried in the air-bath at 100° for a short time, and then extracted with the acetone mixture.

The success of the separation depends to a great extent upon the conditions under which the filtration of the valerate from the undissolved acetate is carried out. After many experiments I have found the following method to give the best results: A funnel is taken with a very long stem, which is passed through a cork in a small inverted bell-jar, filled with hot water for the purpose of keeping the solvent near its boiling-point during the process. A small plug of glass-wool is inserted in the upper part of the stem, and is then covered with a thin layer of fine asbestos, which should allow the solution to pass rapidly through. When the extraction is thought to be complete, it is well to dry the residue in the flask in an air-bath for a few minutes, and to treat it with a further small quantity of the solvent. Owing to the slight solubility of the valerate in the acetone at temperatures much below its boiling-point, it is essential that it should be kept nearly at this temperature throughout the process.

The following results were obtained:

Taken.			Found.		
Acetic Acid.	Valeric Acid.	Total.	Acetic Acid.	Valeric Acid.	Total.
0·102 gr.	0·087 gr.	0·189 gr.	0·104 gr.	0·084 gr.	0·188 gr.
0·102 gr.	0·087 gr.	0·189 gr.	0·105 gr.	0·081 gr.	0·186 gr.
0·102 gr.	0·087 gr.	0·189 gr.	0·103 gr.	0·084 gr.	0·187 gr.
0·157 gr.	0·044 gr.	0·201 gr.	0·159 gr.	0·041 gr.	0·200 gr.
0·153 gr.	0·043 gr.	0·196 gr.	0·155 gr.	0·040 gr.	0·195 gr.

The best "commercially pure" acetone should be purchased, and allowed to stand over a considerable quantity of dry chloride of calcium for one or two days, that portion which boils constantly at 56° being separated and used for the preparation of the acetone solution.

The valeric acid determination is almost invariably a little low, owing to the impossibility of removing the last trace from the acetate by washing. This can always be detected by the odour, when sulphuric acid is subsequently added to the acetate.

My thanks are due to my assistant, Mr. Dick, for the help he has given me in connection with this work.

Discussion.

The President (Dr. Dyer) said the process was one that would possibly be useful in the determination of small quantities of amyl alcohol in spirits. In the

method brought before the Society many years ago by Dr. Dupré, the amyl alcohol was fractionated from the spirit, and obtained ultimately along with some ethyl alcohol. It was oxidized, neutralized with barium carbonate, and, in the barium salt thus formed, the barium was determined, from which the valeric acid was calculated.

Mr. RICHMOND observed that ordinary fusel oil was a mixture of about equal parts of the active amyl alcohol and the iso-amyl alcohol, and would give a corresponding mixture of the two valeric acids on oxidation. It would be interesting to know whether or not the active valeric acid behaved in the same way as the iso-valeric acid. Perhaps the author had made some experiments that would yield information on this point.

Mr. HEHNER said he supposed that, as the molecular weight advanced, the sodium salt would become more and more soluble. He inquired whether, in applying the method to other separations, the author would consider it necessary to use 99·5 per cent. acetone, or whether some weaker strength might be used; and also how a lower acid, such as butyric acid, might be expected to behave under a similar process.

Mr. BEVAN asked how the author managed to hit upon 99·5 per cent. acetone, and whether the fractional percentage of water was essential.

Mr. CHAPMAN said that want of time had prevented his going beyond the immediate object of the investigation, which was simply the separation of acetic and isovaleric acids, so that he could not at present say anything as to the behaviour of active valeric acid, or of any lower acid. He intended, however, to continue the investigation, and to ascertain to what extent the process might be employed in determining fusel oil. Mr. Hehner's suggestion as to the increased solubility of the sodium salt with the rise in molecular weight was doubtless correct. With regard to the solvent, he thought it probable that, in other separations, other strengths of acetone might be employed. It seemed curious that the presence of such a very small quantity of water should have such a marked effect; but sometimes, he thought, insufficient importance was attached in analytical procedure to the influence on solubilities exerted by a small quantity of some third substance.

CAPER TEA.

By JOHN WHITE, F.I.C.

(*Read at the Meeting on March* 1, 1899.)

IN the course of the last two years I have had occasion to examine a considerable number of samples of tea, particularly of that variety distinguished by the name of caper tea, submitted to me under the Sale of Food and Drugs Act. Prior to this time I had always regarded tea of any description as an article which was sufficiently supervised by the provision made for its analysis and inspection in bond before being distributed for sale. The Local Government Board returns evidence the fact that this arrangement has worked well in the past, for although tea, and especially caper

tea, was found to be largely adulterated in the early days of the Food and Drugs Acts, either by the addition of exhausted or foreign leaves, or by the introduction of a comparatively large proportion of extraneous mineral matter, in the year 1897, of 403 samples of tea analysed by the Public Analysts of England and Wales, only one was certified to be adulterated. Further, it was stated in the Local Government Board Report for 1897, that of 4,289 samples of tea analysed under the Act during the ten years ending with 1896, only four were condemned.

In the ANALYST for the present year (vol. xxiv., p. 30) a paper appears by C. Estcourt, in which he emphasizes the fact of the disappearance of adulterated caper tea from the retail market, and quotes a report from the Government laboratory containing the results of analysis of seven samples of caper tea, described as authentic samples out of bond.

The following table gives the results obtained by me from samples of caper tea submitted under the Act between June, 1897, and January, 1899. These samples were not confined to any one particular district, but were collected from various parts of the county of Derby. The estimations were made upon the undried tea, and in nearly every instance the figures given represent the mean of three or four separate determinations. The results are expressed in parts per cent.:

TABLE.

Number of sample.	1.	2.	3.	4.	5.	6.	7.	8.
Total ash	12·20	11·34	11·31	9·90	9·88	13·47	8·80	8·82
Ash soluble in water ...	2·76	2·79	2·84	3·24	2·98	2·80	2·80	3·20
Ash insoluble in water ...	9·44	8·55	8·47	6·66	6·90	10·67	6·00	5·62
Mineral matter insoluble in acid	5·66	4·66	5·10	4·40	3·84	6·26	3·10	3·10

The mineral matter which remained after boiling with dilute hydrochloric acid, with subsequent thorough washing and ignition, consisted in each instance of clean sharp sand mixed with small stones, among which occurred fragments of quartz. The original mineral matter was not in any sample noticeably magnetic.

I do not apprehend any serious difference of opinion as to whether or not these samples are adulterated; it will, I believe, be generally admitted that the extraneous mineral matter shown by these figures is present in too high a proportion to have been "unavoidably mixed in the process of collection and preparation" of these teas. This is certainly obvious in respect of samples 1 to 6, the results of which will possibly justify the chief purpose of this paper, viz., to show that after having lain dormant for many years, the sophistication of caper tea has again become a practicable process, and the sale of the spurious product has apparently become possible.

Proceedings were successfully instituted against the vendors of samples 1, 2, 3, and 4, and are pending in the case of 6; sample 5 will be referred to later. I advised that no action be taken respecting 7 and 8, and also with reference to several other samples containing a total ash of slightly over 8 per cent., as I thought it distinctly undesirable that prosecutions should be undertaken unless the adulteration proved to be of substantial extent.

In addition to the eight samples the results of which are set forth in the above table, I have also found nine other samples yielding a total ash ranging from 8 to 8·4 per cent.; the highest percentage of mineral matter insoluble in acid present in these was 2·7, which occurred in one sample, the next highest being 2·56. Some years ago this Society fixed as a limit 8 per cent. as a maximum of total ash to be permitted in genuine tea, when calculated upon the tea dried at 100° C. Were this figure rigidly adopted, these nine samples must perforce be condemned, but this, in my opinion, is a standard that should be interpreted with very considerable discretion.

Some fifty further samples yielded a percentage of total ash varying from 6·2 as a minimum to 7·9 as a maximum, the average being 7·04, and, what I think is especially important, in no instance did the mineral matter insoluble in acid exceed 2·0 per cent. These results indicate what may fairly be held to represent the composition of genuine caper tea, though both A. H. Allen and Wynter Blyth have expressed the opinion that this variety of tea hardly deserves the description of "genuine," indeed, the former "believes it to be invariably a factitious article." Whether this be so or not, caper tea has come to be recognised as a distinct article of commerce, and one that is in great demand, more especially by the lower classes and by those tea-drinkers who prefer tea with what is known as a "rough" flavour. Caper tea is also believed to be of considerable value for use in mixed teas for the purpose of flavouring the mixture; sample 4 was sold at the high price of 3s. 8d. per pound, and was said by the vendor to be of especially superior quality and character.

The argument has been raised that it is unfair to purchase caper tea under the Act, inasmuch as it is an article which palpably is adulterated by the tea growers abroad, and that the unfortunate vendor who may be prosecuted here is the innocent victim of circumstances outside his control. It is also argued that to buy this special variety of tea appears to be an attempt to entrap the unwary trader. These arguments do not appear to me to be either logical or sound; the Sale of Food and Drugs Acts were passed primarily with the object of protecting the consumer. The process, moreover, by which caper tea is said to be manufactured, viz., by coating the leaves with gum or starch, and subsequently rolling them up into small granules of rounded shape, is in itself highly suspicious. It seems quite possible that this method was adopted for the purpose of allowing the tea to be loaded with mineral matter, the shape of the finished article being favourable to the concealment of it inside the leaves of the tea. Assuming these conclusions to be correct, it is plain that caper tea should not be allowed to compete unduly with the ordinary kinds of tea.

I would suggest that, having regard to the report already referred to from the Government Laboratory, from which it appears that caper tea containing 2·68 per cent. of mineral matter insoluble in acid (I obtain this figure by adding together the sand and the silica less sand) is regarded as genuine, that the presence of 3 per cent. should be considered by Public Analysts to be an adulteration. It is doubtful, however, in view of the result of the appeal Shortt *v.* Robinson, whether a conviction will be obtained until a percentage of 3·5 is exceeded.

Sample 5 was certified by me to contain at least 3·5 per cent. of mineral matter consisting of small stones and sand, and became the subject of the above-mentioned

appeal. At the hearing of the case before the magistrates I gave evidence, and a newspaper report of the certificate from the Government Laboratory, given in respect of Mr. Estcourt's case at Manchester, was produced by the defendant's solicitor, who, however, neither disputed my analysis nor called any rebutting evidence. Our solicitor objected to the production of this document, on the ground that it was not evidence, but it was read. Eventually the Bench dismissed the summons, but granted a case for the decision of the Court of Queen's Bench. The following is the report of the appeal, taken from the *Times* of February 9 last:

QUEEN'S BENCH DIVISION.

(Before Mr. JUSTICE LAWRANCE and Mr. JUSTICE CHANNELL.)

Shortt v. *Robinson.*

This was a case stated by justices of Derbyshire, sitting at Chesterfield, at the hearing of an information against Mark Robinson for selling caper tea adulterated with 3·5 per cent. of mineral matter, consisting of sand, small stones, etc.

Mr. J. H. Etherington Smith appeared for the appellant, the inspector; Mr. Appleton for the respondent, the seller of the tea.

The information, taken out under the Sale of Food and Drugs Act, 1875, was heard on October 22. These facts were proved.—William Marples deposed to purchasing the tea at the shop of the respondent on behalf of the appellant, W. A. Shortt, the Inspector of Weights and Measures for the northern division of the county of Derby. The tea was divided into three parts for the purpose of analysis. The result of the analysis was that it contained 3·5 per cent. of foreign ingredients. At the hearing the County Analyst was called, and swore to the above. In the course of his cross-examination, he was asked whether he had seen in the *Grocer* newspaper a case heard at the Manchester Police-Court on August 16, in which a Somerset House report showed that extraneous matter was necessarily present in caper tea. The analyst admitted that he had seen the report of the case in a paper, not the *Grocer*. The appellant's solicitor objected to this as evidence. The justices allowed the question, though they held that the report was not evidence. The report in the *Grocer*, however, was attached to the case. The analyst in cross-examination stated that, in the course of sixty-five analyses of caper tea, he had found the amount of sand and stones to be not so much as 2 per cent., and that only six specimens contained as much as 3 per cent. It was contended for the appellant that an adulteration of 3·5 per cent. having been proved, there must be a conviction. The justices found that the purchaser asked for caper tea, and that such tea was well known as one manufactured for the purpose of using in small quantities by way of addition to ordinary tea for flavouring it. The justices also found that it was well known that caper, like ordinary tea, was grown under conditions which afforded unusual chances of the introduction of sand and small stones, the tea being grown on friable, sandy soils, and it being a condition of successful growth that rain should be not only very frequent, but copious. They considered that, having regard to these facts, they were not justified in convicting the defendant on so small a percentage as 3·5 of extraneous matter of a nature peculiarly incident to the growth of the article. Moreover, it appeared from the evidence of the analyst himself that there was necessarily some percentage in caper tea of the extraneous matter complained of. They accordingly dismissed the information. The question for the opinion of the Court was whether the justices were justified in considering, on the facts before them, that a presence of extraneous matter to the extent of 3·5 per cent. did not necessarily constitute an adulteration.

Mr. Smith contended that the justices were wrong. They had evidently acted on the report of the Somerset House authorities set out in the case heard in Manchester. That was

not evidence in the case before them. The analyst might have been asked his opinion of the report. If he had affirmed it, then there might have been something for the justices to act on. He was not asked for that, however. His analysis uncontradicted was sufficient to show adulteration under Section 24. He cited Dargie *v.* Dunbar (21 Scottish Law Reports, 536), Regina *v.* Field and others, Justices of Hampshire (64 L. J., M. C., 158). In the Manchester case the defendant gave evidence that the tea when sold was in the same condition as when it was brought out of bond. Section 30 provided for there being an analysis of tea in bond. There was no such evidence in this case.

The Court dismissed the appeal.

Mr. Justice Lawrance said that, in his opinion, the decision of the magistrates ought not to be disturbed. The analyst had stated that there was stone, sand, etc., to the amount of 3·5 per cent. in the sample of tea submitted to him. His attention was then called to the report of the Somerset House authorities supplied in another case dealing with caper tea generally. The analyst admitted that he had seen the report. An objection was taken to the admission of the report as evidence of the facts. It was not stated in the case that the analyst expressed any opinion on the report. In the result, the magistrates apparently brought their own knowledge to bear on the matter, and without calling on the respondent to say whether the tea was in the same state as when it came to him out of bond, dismissed the information, being satisfied that there were always foreign ingredients present in tea.

Mr. Justice Channell concurred. He thought the Court could not interfere in this case. The justices were not wrong in law, unless it were in receiving something as evidence which was not strictly evidence. But the ultimate conclusion at which they had arrived was one to which they were justified in coming. They must have acted on some knowledge of their own. They were entitled to do so, as could be seen by Regina *v.* Field. He thought, in fact, that they had treated the facts about caper tea as well known, partly, at any rate, because they had read in print in the report from Somerset House that there were usually foreign ingredients present in tea. The result of the finding of the justices was right. They thought that the adulterative matter was so near normal in amount as not to justify them in convicting the respondent. The proper course for them to have followed would have been to ask the complainant whether he would insist on going on if they intimated that, supposing the facts set out in the report were properly proved, they would not convict. In all probability the prosecution in such a case would be given up. The magistrates apparently treated this report as evidence. In that they were wrong, but it was only misreception of evidence at the worst, and nowadays, where the misreception of evidence did not affect the result, especially where there had been an acquittal, the Courts would not interfere. The appeal must be dismissed.

The result of this appeal shows once again how far-reaching in its effects a report from the Government Laboratory may be, but the process of reasoning by which the conclusion is arrived at—that because a given sample of authentic caper tea contains 2·68 per cent. of a foreign ingredient, another sample containing 3·5 (or, as I stated in evidence, 3·8 per cent.) is also genuine—does not commend itself to me. I am decidedly of opinion that 3 per cent. as a maximum of foreign mineral matter to be present in caper tea is fair and reasonable, and that if it exceeds that figure the tea should be condemned. There does not appear to me to be any good and sufficient reason why an amount of foreign material that would not for a moment be tolerated in ordinary black or green tea, should be held to be lawful and proper when occurring in caper tea.

ON SOME ANALYSES OF GINGER

By E. G. Clayton.

(*Read at the Meeting, Wednesday, March* 1, 1899.)

Recently I had occasion to examine a considerable number of samples of commercial ginger, and the results of the analyses of these samples are stated in the following table, in which there is a rough classification, according to the origin, condition, and quality of the ginger. It is believed that the series is fairly representative, the samples having been supplied to, or obtained for, me by well-known spice-dealers. Time did not permit me to carry out a fuller investigation; but the analyses for the most part comprise about a dozen estimations: soluble and insoluble ash; the extracts dissolved by cold water, ether, absolute alcohol (after complete extraction with ether), and petroleum spirit; essential oil; alkalinity, calculated as potash, and chlorine, in the soluble ash; and, in some cases, the ash, alkalinity of the ash, and chlorine, in the cold-water extract. Water and silica were also determined in certain instances. All the series of estimations may be regarded as strictly comparable.

The main objects of the inquiry were: (1) To form an opinion whether certain samples submitted to me were, or contained any admixture of, "spent" ginger; (2) to ascertain whether any further information could be amassed as to the effects on the composition of ginger of the operation known as "washing"; (3) to study the influence on the analytical figures of the commercial processes of *grinding*, with removal of the fibre, and *scraping or cutting off the surface* of the rhizome—this latter operation being alleged to be resorted to with the view of giving the ginger a better appearance; (4) to collate analytical data from samples of different geographical origins, and in various conditions, as whole, ground, washed, or otherwise; and (5) to compare the figures yielded by different market qualities (as indicated by the prices) of the same variety of ginger.

In the table (which includes the results of analyses of thirty-seven samples) five principal series are included: (A) Whole gingers, washed and unwashed; (B) commercially-ground gingers; (C) spent gingers; (D) a series of four samples of the same Cochin ginger at different stages of grinding, etc.; and (E) samples of similar ginger, washed, scraped, and cut, as well as some of the cuttings and shavings removed from the surface.

Included in the table are the results of an analysis of green ginger (F), and of a few estimations made in some samples of whole Cochin ginger (G), previously steeped in the laboratory for forty-eight hours in water and in diluted alcohol. The averages and limits observed are also stated.

I desire to express my acknowledgment of the help cheerfully rendered by my assistant, Mr. Henry H. Williams, in the course of these analyses.

The figures obtained, of course, in the main confirm those of other observers, but two or three points may be touched upon.

In the estimations of moisture, it was noticed that after prolonged drying in the water-oven many of the samples began to gain in weight, this being due, I suppose, to oxidation. Hence it was rather difficult to arrive at constant weights, but the lowest of a series of weighings was taken in each instance.

The effect of washing in diminishing the percentages of some of the constituents, such as the ash, soluble ash, cold aqueous extract, etc., is clearly shown; and a similar influence appears to be exerted to a certain extent by grinding, removal of the fibre, and the operation of cutting or scraping the exterior of the rhizome. But the figures have also shown that what I at one time thought possible is not the case; namely, that by the combined effect of all these commercial processes—washing, scraping, cutting, grinding, and separation of fibre—the composition of genuine ground ginger might be so altered or reduced as to approximate to that of spent ginger. Some of the results obtained clearly indicate, at all events as regards Cochin ginger, that even after all these operations have been carried out, the percentage proportions of many, if not most, of the constituents remain sensibly—indeed, considerably—above the corresponding numbers found in spent ginger. I do not know what might prove to be the case with Japan ginger; the figures do not go far enough to show.

The above conclusion is opposed to my earlier anticipations and belief; but some of the later results, notably the figures obtained with the samples constituting series E in the table, have been entirely convincing.

The determinations of the ethereal extract and alcoholic extract (after removal of everything soluble in ether) were effected in a Soxhlet apparatus, the first occupying from two to three days, the second from three to four days. The alcoholic extracts appear to be less than those obtained by other observers; but the last alcohol extraction-fluid was in a number of cases tested, and found to leave practically no residue. Moreover, as it seemed possible that in some instances the extraction with ether had not been carried far enough (in spite of the long time it occupied), and that some matter had been left behind, to be removed by the absolute alcohol, an experiment was made to test this point. Duplicate estimations of the two components were made in one of the samples, absolute alcohol being used *first*, the extract dried to constancy, weighed, and *then* treated with ether. The resulting ethereal extract was dried until constant, and weighed. The numbers obtained were:

Alcoholic extract	0·65 per cent.
Ethereal ,, ...	3·73 ,,

By the other process (using ether first and alcohol last) the same sample (No. 30) had yielded:

Alcoholic extract (after ether extraction) ...	0·75 per cent.
Ethereal extract	3·55 ,,

The fair agreement of the two pairs of estimations would seem to point to the substantial accuracy of the rest of these determinations, and to indicate that the

matter remaining for extraction by absolute alcohol, after ethereal treatment, may be somewhat lower than is supposed, *provided* that there has first been complete extraction with ether.

It will be seen that the fixed extract removed by petroleum spirit was found to be a tolerably constant figure in the genuine samples, and an attempt was made to determine approximately in each case the essential oil of ginger, by a process somewhat akin to that originally described by Dr. Thresh, namely, extracting the ginger with ether, air-drying and weighing the extract, and then drying in the water-oven to constancy; but it was thought possible that the method might be advantageously modified by the substitution for ether of petroleum spirit (by which less of the resinous and other constituents of ginger are extracted than by most other solvents). The figures resulting from this modified process are decidedly lower than those previously obtained, and obviously very variable. Of course, the method is necessarily imperfect.

Chlorine was found to be practically absent from the soluble ashes of the samples of spent ginger; the two exceptions at the end of the series of spent ginger samples I believe to have been, not exhausted ginger only, but to have contained some admixture of unexhausted ginger. Curiously enough, very small quantities of chlorine appeared in most of the ashes of the cold aqueous extracts of the spent ginger samples. Possibly this might be explained by the presence of some soluble organic compound containing chlorine.

The samples Nos. 15*, 16*, 17* and 18*, were different market qualities of the same kind of ginger, namely, Cochin ginger; it is remarkable that the proportions of the principal constituents diminish as the price increases; and the question arises whether the real quality of a sample may not sometimes be sacrificed to mere appearance—whiteness, and so forth.

Discussion.

The President (Mr. Fisher) said it seemed to him that washing ginger till all its valuable constituents disappeared was but little different from mixing it with exhausted ginger, seeing that the result was the same in both cases. It appeared possible, however, that excessive washing was allowed to take place through ignorance on the part of those engaged in the trade.

Mr. Allen desired to ask what figures the author would take as minimum limits in genuine ginger for soluble ash and for cold water extract, the latter determined directly on the ginger, and not after treatment with alcohol and ether. If ginger was exhausted as completely as possible with water, it would still yield an appreciable proportion of soluble ash. He would like to hear if the author had any information as to the limits of silica in exhausted ginger. This constituent was occasionally present in very considerable proportion. He was much interested in the specimens of exhausted ginger free from chlorides. Mr. Stock and other analysts, and he (Mr. Allen) himself to a limited extent, had determined, not the alkalinity, but the actual potash in the soluble ash of ginger and other plant-products, a factor which sometimes yielded valuable information. His practice, he might mention,

was to estimate soluble ash both by difference and by direct determination by evaporation; which latter method generally gave the higher result.

Dr. DYER said that he had recently analysed, in connection with a case in which both he and the author had been interested, a series of samples which he believed were duplicates of many of those referred to in the paper. His conclusions were in agreement with those of the author. The question had been raised at the time by a member of the ginger trade, whether the paring of ginger might not affect the results of its analysis, and particularly the percentage of soluble ash. He (Dr. Dyer) had at the moment not felt prepared to say that it might not, for little was known as to the distribution of the salts contained by the root. The samples which he and the author had both analysed consisted of Cochin ginger of various kinds and in various stages of manufacture — pared and unpared, parings, and fibre separated during grinding. The results of the analyses indicated that any legitimate process to which the ginger might be submitted produced practically no variation in its composition, that is to say, no such variation as could give rise to a suspicion of admixture with spent ginger. One of the samples approximated in composition to what might be regarded as partially exhausted ginger, but that sample had been submitted to a process of excessive washing. There was no evidence that properly-conducted washing resulted in any approach to what might be called exhaustion. It was, however, an undoubted fact that some ginger-washers—for ginger-washing was a trade—steeped the ginger in hydrochloric acid for the purpose of removing lime from its surface; and it was obviously necessary to afterwards steep the ginger in water to get rid of the acid. It seemed to him that no sample so treated could properly be regarded as unexhausted, seeing that such washing largely deprived it of both strength and flavour, and so partially exhausted it. He thought that the discussion at present going on in the trade on this subject would have the effect of altogether stopping the washing of many kinds of ginger that were now quite unnecessarily washed.

Mr. CLAYTON said it seemed rather difficult on the spur of the moment to lay down limits which would carry authority. He thought he might suggest that 1 per cent. would about represent the lowest limit of soluble ash in genuine ginger, but he only made the suggestion tentatively and with diffidence. In a sample which he had been assured was genuine, but which was of unknown origin, he had found 1·43 per cent. of soluble ash. That was the lowest figure for soluble ash that he had actually met with in genuine ginger, apart from one sample of washed Japan ginger. He quite agreed with Mr. Allen as to the difficulty of getting out all the soluble ash by exhaustion. This not only applied to treatment with water, but also with alcohol and ether. He was unable to express any opinion as to silica, not having included this point in his investigations; nor had he determined the actual potash present, as he was aware had been done by Mr. Stock. He might mention that the prices of four of the samples referred to in the paper—viz., Nos. 15, 16, 17, 18—were respectively 90s., 50s., 40s., and 28s. per cwt., and it was curious to note that, as the price rose, the total ash, soluble ash, cold-water extract, and most of the other constituents diminished.

ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

FOODS AND DRUGS ANALYSIS.

The Examination and Characteristics of Commercial Fennel Seed. A. Juckenack and R. Sendtner. (*Zeit. Untersuch. Nahr. Genussm.*, 1899, ii., 69-75.)—The authors have examined a large number of the principal kinds of fennel seed found in the German market, viz., German, Italian, Macedonian, and Galician, with the object of obtaining data for the detection of exhausted and coloured seeds.

The moisture, mineral matter, aqueous extract, alcoholic extract, and the earthy impurities and mineral constituents corresponding with these, were determined on specimens of each variety with the following mean percentage results:

Fennel Seed.	Water.	Total Mineral Matter.		Mineral Matter Deducting Soil.		Aqueous Extract.		Alcoholic Extract.	
		Natural Substance	Dry Substance	Natural Substance	Dry Substance	Natural Substance	Dry Substance	Natural Substance	Dry Substance
German ..	13·85	8·027	9·263	7·966	8·845	22·21	25·66	11·16	13·21
Italian ..	11·74	8·883	10·065	7·987	9·056	23·87	27·05	14·90	16·85
Macedonian	9·88	12·520	13·890	8·010	8·880	26·11	28·96	14·56	16·16
Galician ..	11·59	12·001	14·253	7·844	8·849	21·62	24·46	14·27	15·96

The following table shows the effect of steam extraction on the composition of the seed:

	Water.	Mineral Matter.		Aqueous Extract.		Alcoholic Extract.	
		Natural Substance.	Dry Substance.	Natural Substance.	Dry Substance.	Natural Substance.	Dry Substance.
Saxon fennel ...	12·09	7·445	8·480	21·10	24·00	10·24	13·22
do. extracted	7·74	5·444	5·900	6·40	6·93	4·23	4·58

In order to detect the presence of exhausted seeds, from 3 to 5 c.c. of the fennel are shaken in a reagent-cylinder with from 3 to 4 volumes of 96 per cent. alcohol, and allowed to stand for a short time. The extracted seeds become dark or black, while the genuine seeds retain their original colour. The supernatant alcohol varies from dark green to pale green according to the proportion of extracted seeds in the mixture.

A second test consists in mixing from 2 to 3 grammes of the seed with 20 c.c. of water in a small basin, and allowing the mixture to stand, when the genuine seeds float (on account of their higher percentage of fat, which repels the water) and retain their colour, while the extracted seeds become dark and fall to the bottom.

A difference can also be observed under the miscroscope, the oil-streaks in the genuine fennel being more or less refractive to light, instead of, as in extracted seeds, being dull and black. The ribs of extracted seeds are browner in colour and less sharp in outline than untreated seeds.

The authors conclude from their analyses that the following limits may be taken as the lowest for genuine fennel seed:

	German. Per cent.	Italian. Per cent.	Macedonian. Per cent.	Galician. Per cent.
Moisture	11·5	10·0	9·5	10·0
Mineral matter calculated on the dry substance (excluding earth)	8·0	8·0	8·0	8·0
Aqueous extract (on dry substance)	23·5	24·0	24·0	24·0
Alcoholic extract (on dry substance)	12·0	13·5	13·5	14·5

They also consider that pure undamaged fennel should contain at least 70 to 80 per cent. of grains capable of germinating. Artificial colour is sometimes used to conceal the extraction of the seeds or to improve their appearance. It may be detected by the following tests: (1) When examined under the microscope the ribs of uncoloured seeds are seen to be sharply differentiated from the depressions, which is not the case with coloured seeds. (2) When 5 c.c. of the coloured fennel are shaken for a minute with 25 c.c. of 96 per cent. alcohol, the liquid is at first turbid and green, but on standing the colouring matter is deposited. (3) The colour may be isolated by washing the seeds with water, then shaking them with absolute alcohol, allowing the colouring matter to settle and filtering it off.

The only colouring matter which the authors have met with is ochre. They have not, like Neumann-Wender, detected chrome yellow in any coloured seeds.

C. A. M.

Dionine. L. Hesse. (*Pharm. Central H.*, 1899, xl., 1; through *Chem. Zeit. Rep.*, 1899, 32.)—This new therapeutic agent consists of the hydrochloride of mono-ethylmorphine. The base forms brilliant prisms, having the formula $C_{17}H_{17}NO.OH.OC_2H_5 + H_2O$. The hydrochloride is a white, odourless, bitter, micro-crystalline powder, which melts and decomposes at 123° to 125° C. It is easily soluble in water, giving a neutral solution, and is chemically and physiologically similar to codeine, being exhibited in the same doses. If 5 c.c. of a 10 per cent. solution of codeine hydrochloride are treated with a few drops of 0·910 ammonia, the alkaloid is thrown down; but 1 c.c. of alkali is sufficient to redissolve the precipitate permanently. With dionine, 5 c.c. of ammonia first produce a clear liquid, from which the base gradually separates in crystals. Dionine may be distinguished from morphine by a solution of potassium ferricyanide containing a trace of ferric chloride; when 1 c.c. of a 1 per cent. solution of morphine is added, the liquid becomes dark-blue immediately; dionine only slowly bluish-green.

F. H. L.

The Assay of Liquid Extract of Ipecacuanha. H. Wilson. (*Amer. Journ. Pharm.*, 1899, lxxi., 73-77.)—The author criticises the method of determining the alkaloid in this preparation, as given in the British Pharmacopœia of 1898 (p. 115), asserting that it is complex and tedious, and that a considerable proportion of the alkaloids is lost in the lead precipitate. In its place he recommends the following method: 20 c.c. of the strong liquid extract are diluted with an equal volume of water, and the alcohol removed by evaporating the mixture on a water-bath to less than half its volume. After the addition of 1 c.c. of sulphuric acid, the liquid is transferred to a separating funnel (the dish being washed out with 20 c.c. of water) and extracted three times with 10 c.c. of a mixture of equal volumes of chloroform and

ether, the extracts being rejected each time. The aqueous residue is then rendered alkaline, and again extracted three times with the same quantity of mixed ether and chloroform. The united extracts are evaporated to dryness in a weighed dish, and the residue dried below 80° C. and weighed.

The following table gives the results obtained by this method in comparison with those yielded by the officinal process:

Ipecacuanha Extract.	Officinal Process.			New Process.
	Alkaloids extracted. Gramme.	Alkaloids lost in Lead Precipitate. Gramme.	Total Alkaloids. Gramme.	Alkaloids found. Gramme.
1.	0·386	0·031	0·417	0·417
2.	0·393	0·028	0·421	0·426
Mean	0·389	0·029	0·419	0·421

The author considers that at present the alkaloids of ipecacuanha cannot be accurately determined by titration. For, if it be assumed that the alkaloids consist of emetine with molecular weight 248 and cephaëline (molecular weight 238), in about equal proportions, each c.c. of decinormal acid should correspond with 0·0241 gramme of the mixed alkaloids. But in practice the results obtained by a volumetric estimation using this factor are from 50 to 60 milligrammes lower than the results of the gravimetric methods. This, in the author's opinion, is probably due to the presence of the third alkaloid, which Paul and Cownley believe to have a higher molecular weight that either of the others. C. A. M.

TOXICOLOGICAL ANALYSIS.

The Isolation and Identification of Sulphonal in Forensic Work. C. Kippenberger. (*Zeit. Untersuch. Nahr. Genussm.*, 1899, ii., 75-91.)—I. *Isolation of Sulphonal.*—The usual method of extracting sulphonal from cadaveric remains consists in extracting the substance with hot alcohol, evaporating the extract, dissolving the residue in hot water, filtering, evaporating and purifying the residue by crystallization from boiling alcohol. According to the author, the preparation thus obtained may be so contaminated with organic impurities as to render the identification of sulphonal uncertain.

From his investigations it appears that 100 c.c. of benzene (B.P. 80°-82° C.) dissolve 8·01 grammes of sulphonal, 100 c.c. of petroleum spirit (B.P. 30°-50° C.) 0·06 gramme, and 100 c.c. of chloroform 32·5 grammes. Its solubility in other solvents is: Water, 500 parts; boiling water, 15 parts; ether, 133 parts; alcohol, 65 parts; boiling alcohol, 2 parts. Toluene and acetone dissolve it in considerable quantities, and carbon tetrachloride dissolves it sparingly.

The following method of quantitative isolation is recommended when sulphonal has been detected qualitatively. The dry or only slightly moist material is extracted with chloroform or benzene on the water-bath, and the extract filtered and evaporated. The residue is treated several times with small quantities (2 to 4 c.c.) of petroleum spirit, which removes fat and colouring matter, and leaves the sulphonal as a white mass. If cholesterin was present in the original substance, it is separated from the sulphonal by treatment with water, and extracting the sulphonal from the

aqueous solution with chloroform or benzene. This treatment can also be used to separate sulphonal from the xanthin bodies, which are only sparingly soluble in water. The method of isolation and purification also effects a separation from these bases, owing to their insolubility or sparing solubility in benzene, and their fairly ready solubility in petroleum spirit.

When putrefactive decomposition compounds are simultaneously present, the author's method of extracting alkaloids with glycerin tannic acid solution (ANALYST, xx., 201) often gives satisfactory results. If vegetable alkaloids have been also isolated, they can be readily separated by precipitating them from an acid solution by means of iodine in potassium iodide.

It should not be lost sight of that fatty oils dissolve a considerable proportion of sulphonal on heating, which they only deposit very slowly on cooling, although in the cold they only take up slight traces.

In the method described by Hilger and Tamba (*Arch. d. Pharm.*, 1887, 225, 408) for the separation of ptomaines and alkaloids by means of oxalic acid solution, the sulphonal remains permanently dissolved.

II. *The Identification of Sulphonal.*—Sulphonal, or diethyl-sulpho-dimethyl-methane $(CH_3)_2C(SO_2C_2H_5)_2$, forms odourless crystals, which are neutral in reaction, and to most people tasteless. It is unacted upon by cold concentrated sulphuric acid, by boiling concentrated nitric acid, by bromine, or by boiling solutions of alkaline hydroxides. It melts at 125° to 126° C., and volatilizes at 300° C with slight decomposition.

Several tests for its identification have been based on the formation of strong odours attributed to mercaptan, on treating it with different substances. Thus, Vulpius heats it with twice its weight of potassium cyanide ; Ritsert uses pyrogallol ; Scharz, charcoal ; and Strobel, zinc chloride.

When sulphonal is heated with twenty times the amount of dry sodium acetate, mercaptan and hydrogen sulphide can be detected in the decomposition products.

The author has attempted to estimate the mercaptan in this reaction by absorbing it with a solution of silver nitrate, and collecting the silver mercaptide, but with only partial success.

When sulphonal is heated with magnesium powder, a sublimate is obtained which consists of malodorous sulphides of various composition, with traces of oily thioalcohols which solidify to a crystalline mass on cooling.

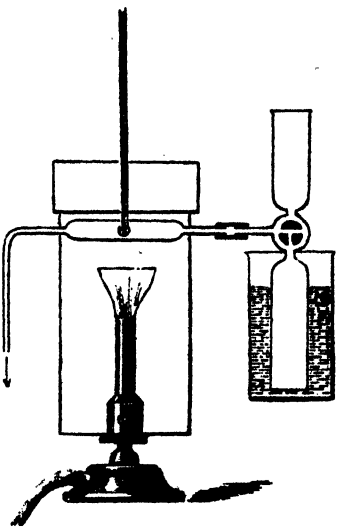
On heating a mixture of manganese peroxide and sulphonal, white acid vapours with a penetrating smell are obtained, and the sublimate is a perfectly white substance with no trace of the yellow oil formed in the magnesium powder reaction. A similar reaction is obtained with lead peroxide, barium peroxide, platinum black, and alkaline nitrates, with the formation of oxidation products of the thioacetones derived from the decomposition of the sulphonal (thioacetone oxides).

When a mixture of sulphonal and an alkaline hydroxide is heated in a silver basin until the mass assumes a reddish tint, and then cooled, the residue on extraction with water and filtration furnishes a solution which becomes dark on treatment with ferric chloride, reddish-violet with sodium nitro-prusside, and on acidification deposits sulphur.

C. A. M.

ORGANIC ANALYSIS.

Estimation of Hydrogen and Methane by Fractional Combustion with Copper Oxide. E. Jager. (*J. Gasbeleucht.*, 1898, xli., 764; through *Chem. Zeit. Rep.*, 1898, 335.)—The processes for separating methane from hydrogen by fractional combustion with air suffer from the disadvantage that, owing to the limited size of the burette, only small volumes of the gas can be taken for analysis. The author finds that if a mixture of hydrogen, methane, and nitrogen is passed two or three times over copper oxide heated to 250° C., as indicated by the thermometer, the bulb of which is in contact with the tube, the hydrogen alone is oxidized; and by raising the temperature to a red heat, after removing the thermometer, the methane can afterwards be burnt. Small corrections are necessary for the alterations in volume of the oxide during reduction, and for the amount of atmospheric oxygen in the combustion tube; but these can easily be ascertained. The apparatus required is partly illustrated in the accompanying sketch.



F. H. L.

The Determination of Arsenic in Glycerin. A. C. Langmuir. (*Journ. Amer. Chem. Soc.*, xxi., 133-136.)—In this modification of the Polenske method 15 to 20 grammes of crude soap glycerin, or 15 to 20 grammes of saponification- or C. P. glycerin are diluted to 100 c.c. with hot water, and carefully boiled down with 200 c.c. of concentrated nitric acid and 12 c.c. of concentrated sulphuric acid until sulphuric acid fumes are copiously evolved, the nitric acid being finally expelled by strongly heating. The cooled liquid is diluted to 50 c.c. and run slowly into a Marsh apparatus, drop by drop, the flow being regulated so that not more than two bubbles of gas per second pass the lead nitrate wash-bottle. Any escape of undecomposed hydrogen arsenide can be detected by placing a strip of paper, soaked in mercuric chloride, in the further end of the heated tube. The mirror section of the tube is finally separated from the rest, and weighed before and after the removal of the mirror. The arsenic content in saponification-glycerins averaged 0·00018 to 0·00030 per cent., whilst crude soap-lye glycerins gave 0·00064 to 0·01 per cent.

The Gutzeit method gives fairly good indications for comparison with known quantities when mercuric chloride is employed as the detecting agent, the limit of delicacy being about 0·00010 per cent., but the silver nitrate used by Gutzeit gives very imperfect results. C. S.

The Flash-Point of Dilute Alcohol and other Organic Bodies. P. N. Raikow. (*Chem. Zeit.*, 1899, xxiii., 145.)—The author suggests that a determination of the flash-point of inflammable organic substances in the ordinary Abel apparatus may frequently be of service in judging their degree of concentration or of purity.

For instance, absolute ethylic alcohol flashes at 12° C.; addition of 0·5 per cent. of ether reduces it to 9°; presence of 2 per cent. of ether lowers the flash-point to 2·5° C.; so that even 0·1 per cent. may be recognised and estimated. The introduction of 1 per cent. of benzene to monochlorobenzene causes the flash-point to fall from 27·5° to 24°; so that 0·1 per cent. may also be estimated in this case. The operation is carried out exactly as in testing petroleum, but the water-bath and annular air-jacket of the Abel apparatus should be filled with hot, warm, or ice-cold water, air, snow, etc., according to the height of the flash-point expected. Further experiments are in progress; in the meantime the author submits a curve and table of the flash-points of diluted ethylic alcohol. These show that the rise is not in proportion to the amount of water, that there is no break in the curve at the point where maximum contraction occurs (51·9 v/v spirit), as should happen if a definite hydrate were formed, and that alcohol flashes until it contains 97 v/v of water.

FLASH-POINTS OF AQUEOUS ALCOHOL.

Barometer 710-713 mm.

Alcohol v/v.		Flash-point.		Difference per 5 v/v.	Alcohol v/v.		Flash-point.		Difference per 5 v/v.
100	...	12			35	...	27·75		
98	...	13·25	...	2·5	30	...	29·5	...	1·75
96	...	14			25	...	33·25	...	3·75
94	...	15			20	...	36·75		3·5
92	...	15·75	...	2	15	...	41·75	...	5
90	...	16·5			14	..	43		
85	...	17·75	...	1·25	13	...	44·25		
80	...	19	...	1·25	12	...	45·75	..	7·25
75	...	19·75	...	0·75	11	...	47		
70	..	21	...	1·25	10	...	49		
65	...	21·25	...	0·25	9	...	50·25		
60	...	22·25	...	1	8	...	52·5		
55	...	23	...	0·75	7	...	55	..	13
51·9	...	23·75			6	.	58·25		
50	...	24	...	1	5	...	62		
45	...	24·75	...	0·75	4	..	68		
40	...	26·25	....	1·5					

F. H. L.

The Separation of Unsaturated Fatty Acids from one another. K. Farnsteiner. (*Zeit. Unters. Nahr. Genussm*, 1899, ii., 1-27.)—(*a*) *Experiments on the Separation of Oleic Acid as Lead Elaidate.*—Various methods of converting oleic acid into elaidic acid were tried, and it was found that the largest yield could be obtained by saturating the fatty acid with nitric oxide, and converting this into nitrogen peroxide by introducing oxygen. The most favourable conditions were with the temperature at 10° to 20° C., and with 20 to 25 c.c. of nitric oxide to each gramme of oleic acid. A special apparatus is described, in which the reaction was carried out in a flask from which the larger proportion of the air was first removed. The acid obtained was hard, dry and crystalline, melted at about 43° C., and contained from 82 to 86 per cent. of pure elaidic acid.

The lead salt of the impure elaidic acid behaved like those of the saturated fatty

acids as regards its solubility in ether and benzene, 100 c.c. of the former dissolving 0·0086 gramme, and 100 c.c. of the latter 0·0040 gramme, at 16° to 20° C.

The author made use of this fact to determine the amount of acid not converted into elaidic acid in his experiments with pure oleic acid, the lead salts being treated with benzene in the method described in his paper on the separation of saturated from unsaturated fatty acids (ANALYST, xxiii., 285). Although so far he has been unable to obtain sufficiently constant results with the elaidic reaction for a quantitative method to be based upon it, he considers that, applied in the method described, it is a valuable qualitative test for the presence of oleic acid, and for determining the purity of that acid.

(*b*) *Experiments on the Estimation of Oleic Acid as a Barium Salt.*—Dry barium oleate is insoluble or dissolves only in traces in hot benzene. On adding absolute alcohol to the benzene and boiling, the salt is only dissolved with great difficulty; but with weaker strengths of alcohol it is readily dissolved, and is deposited as a crystalline powder on cooling. The separation of barium oleate from benzene (containing alcohol) is so complete that it appears suitable for the quantitative separation of oleic acid from other unsaturated fatty acids whose barium salts are soluble in benzene, etc.

The following experiment with olive oil illustrates the general method employed: About 1·3 gramme of olive oil was saponified, and the hot soap solution precipitated with barium chloride. The precipitate was washed with water, and dissolved in 50 c.c. of benzene containing 2·5 c.c. of 95 per cent. alcohol. The next day the precipitate was filtered off, and dissolved in 50 c.c. of benzene containing 10 c.c. of the alcohol, the precipitate from this being recrystallized from 50 c.c. of benzene containing 20 c.c. of alcohol. The filtrates yielded soluble barium salts containing 0·190 gramme of fatty acids, corresponding to 14·9 per cent. of the oil. The fatty acids recovered from the insoluble barium salts were converted into lead salts and separated into saturated fatty acids (9·95 per cent.) and oleic acid (70·9 per cent.) by the author's process (ANALYST, xxiii., 285). The oleic acid yielded a firm elaidic acid, melting at 41° to 42° C. The saturated fatty acids melted at 57° C.

Earthnut oil contained 30·3 per cent. of fatty acids forming soluble barium salts. A lard with an iodine value of 49·0 gave the following results: Solid acids, 42·2; oleic acid, 39·2; and fatty acids forming soluble barium salts, 13·9 per cent. Cocoa-butter was found to contain: Solid acids, 59·7; oleic acid, 31·2; and other acids, 6·3 per cent.

In the case of butter-fat, cotton-seed oil, and sesame oil, the author was unable to separate the oleic acid in an approximately pure condition by this method, a considerable proportion of the barium oleate remaining dissolved in the benzene, while a corresponding amount of the barium salts and other acids were found in the barium oleate precipitate.

By treating the liquid fatty acids of butter-fat with nitrogen peroxide, as in *a*, about 45 per cent. of elaidic acid was obtained, from which result the amount of oleic acid in the fat was calculated to be about 20 per cent.

The barium salts of the fatty acids can be prepared directly from the fats by saponifying them with a solution of barium hydroxide in equal volumes of benzene and methyl alcohol.

(*e*) *The Detection and Quantitative Estimation of Linolic Acid.*—A method of determining linolic acid is based on the insolubility of linolic tetrabromide in cold petroleum spirit, the precipitate being collected and weighed as described by the author in his former paper (ANALYST, xxiii., 286).

The results thus obtained under varying conditions of bromination (temperature − 10° to + 20° C.) with different oils and fats are summarized below.

Cotton-seed oil total fatty acids yielded a tetrabromide melting at 113° to 114° C. (uncorr.), the amount corresponding with (1) 18·9; (2) 18·45, and 18·2 per cent. of linolic acid. The quantity obtained from the liquid fatty acids in two determinations corresponded with 23·0 and 23·9 per cent., or 17·2 per cent. and 18·2 per cent. respectively on the total fatty acids.

Sesame oil (with 12·1 per cent. of solid acids) was found to contain in its total acids (1) 16·4, and (2) 15·2 per cent. of linolic acid. A second sample extracted in the laboratory gave the following results: Solid acids, 14·1; liquid acids, 86·7; and linolic acid, 12·6 per cent.

Earthnut Oil.—As the total fatty acids yielded no insoluble bromide, the more unsaturated fatty acids were concentrated by treatment of the barium salts with benzene, as in *b*, and the acids recovered from the soluble barium salts brominated in petroleum spirit. The insoluble bromide melted at 113·5, and had a molecular weight of 602·5. The unsaturated acids brominated amounted to about 30 per cent. of the weight of the oil, and the linolic acid obtained to about 6 per cent.

Olive Oil.—This was examined in the same way as the earthnut oil, and the acids from the soluble barium salts treated with petroleum spirit. The bromination products did not dissolve completely in the hot solvent, a powdery residue remaining (probably linolenic hexabromide). The solution deposited crystals, melting at 113° C. on cooling.

Rape-seed Oil.—On brominating the total fatty acids, a considerable quantity of an insoluble substance, of which only traces were soluble in petroleum spirit, but which was much more soluble in ether, was obtained. From its melting-point on crystallization from benzene (178° C.), the author regarded this as linolenic hexabromide.

Almond Oil.—The amount of tetrabromide (M.P. 113-114° C.) obtained from the total fatty acids corresponded with 5·97 per cent. of linolic acid.

Mustard-seed oil gave bromides melting at 178° to 179° C. and 111° to 112° C. respectively. The yields corresponded with 4 per cent. of linolenic acid and 4·5 per cent. of linolic acid.

Butter-Fat.—This was prepared in the laboratory from the milk of cows whose fodder for a long time previously had contained cotton-seed meal. The liquid fatty acids yielded an appreciable quantity of an insoluble bromide which had all the characteristics of linolenic hexabromide. The bromide of linolic acid was not found.

Lard.—Five grammes of the fat were separated by the barium method (*b*), and the acids from the soluble barium salts brominated in petroleum spirit. The bromination product dissolved completely in the hot solvent, but on cooling deposited a small quantity of two bromides, one of which was identified as linolic tetrabromide, while the more insoluble, which melted without purification at 165° was probably linolenic hexabromide.

Horse-Fat.—The liquid fatty acids yielded an insoluble bromide corresponding with 9·9 per cent. of linolic acid in the fat.

Ox-Tallow.—This, like lard, was found to contain a small amount of linolic acid and traces of linolenic acid.

As regards the detection of linolic acid by the formation of its oxidation product (sativic acid), the author points out that Hazura, in all his researches prior to 1888, gave the melting-point of sativic acid as 160° to 162° C., but that, in a paper published in that year in conjunction with Grüssner, the melting-point was given as 173° C. On the other hand, Fahrion found the melting-point of his sativic acid to be 152° C. The author is inclined to believe that Hazura's acid melting at 160° to 162° C. was not pure sativic acid, but contained an isomeric substance. C. A. M.

The Constants of Various Train-Oils. W. Fahrion. (*Chem. Zeit.*, 1899, xxiii., 161.)—The constants of various kinds of fish-oils are collected in the following table. To determine the saponification and Hehner numbers, 2 or 3 grammes of the sample were heated on the water-bath with 10 or 15 c.c. of 2N alcoholic alkali, the spirit removed, the soap dissolved in hot water, placed in a separating funnel, acidified with dilute hydrochloric acid, and when cold extracted with 30 or 40 c.c. of petroleum ether (b. p. 75°), allowing the mixture to rest over-night. The aqueous liquor was drawn off from below, the small proportion of oxidized fatty acids in solution being neglected, and the petroleum ether poured off from above; the insoluble acids washed in warm ether, and the whole of the solvent evaporated on the water-bath, weighing the residue to obtain the fatty acids plus the unsaponifiable matter. This was dissolved in 40 or 50 c.c. of alcohol, and titrated with $\frac{N}{2}$ aqueous potash to yield the "inner saponification number." The neutral liquid was extracted with petroleum spirit, the extracts washed with 60 per cent. alcohol, and dried to give the unsaponifiable matter. By calculation the molecular weight of the fatty acids was thus arrived at. The insoluble oxy-acids in the separating funnel were taken up in warm alcohol, evaporated, weighed, and the ash determined; the difference being the oxidized acids themselves. The sum of the fatty acids, oxy-acids, and unsaponifiable matter gave the Hehner number. The iodine value was estimated with the Waller solution, working for twenty-four hours in the dark, and arranging as accurately as possible for an excess of 50 per cent. of the iodine absorbed—uniformity is particularly necessary in this operation.

It should be noted that when the catch of dorse (Baltic cod) is small, brown liver oils appear on the markets which contain 5 per cent. or more of unsaponifiable matter. This does not always point to adulteration, but simply to a sort of fermentation and the use of a high temperature during extraction of the oils. Such products, of course, are of less value, and 3 per cent. should be taken as the limit. With the exception of herring-oil (No. 9) all were completely liquid and translucent at ordinary atmospheric temperatures, which shows that the greater part of the palmitin had already been precipitated by cold. The colours quoted were those given by a layer 2 cm. thick.

CONSTANTS OF FISH-OILS.

Description.	Colour.	Iodine number.	Acid number.	"Inner saponification number."	Hehner number.	Unsaponifiable matter p.c.	Oxy-acids p.c.	Fatty acids p.c.	Mol. weight of fatty acids.
Medicinal train ...	pale yellow ...	166·2	1·8	181·9	96·49	0·78	0·23	95·48	294·1
Pale dorse ..	yellow ...	162·4	23·5	180·0	96·52	1·30	0·61	94·61	294·1
Brown liver, 1896	brown... ...	140·6	30·9	174·2	95·46	1·50	1·29	92·67	298·2
,, ,, 1897	,, ...	159·8	139·9	174·2	97·05	1·91	1·49	93·65	301·0
Sardine oil ...	yellow ...	191·7	19·2	185·2	95·60	0·48	0·61	94·51	285·7
,, ...	red	167·9	21·7	177·2	96·55	1·01	1·35	94·19	297·7
,, ...	orange ...	160·9	4·6	179·5	97·08	0·63	0·94	95·51	299·5
Stickleback oil ..	yellow ...	162·0	21·6	181·5	95·78	1·73	0·62	93·48	287·4
Herring oil ...	yellow-brown	123·5	44·6	178·5	95·64	0·99	1·59	93·06	291·9
Japan fish oil ...	pale yellow ...	164·0	10·8	185·8	95·52	0·52	1·16	93·84	282·8
,, ,, ...	brown-yellow	157·6	34·2	189·1	96·58	0·67	0·75	95·16	281·7
,, ,, ...	pale yellow ...	135·7	12·3	181·4	97·04	0·82	0·41	95·81	295·7
,, ,, ...	reddish-yellow	108·5	34·5	180·0	96·82	0·86	0·62	95·34	296·6
,, ,, ...	yellow ...	100·1	28·2	183·8	96·51	0·79	0·49	95·23	290·2
Shark oil... ...	pale yellow ...	138·6	8·2	175·1	97·26	1·68	0·18	95·40	305·1
Tunny-fish oil ...	dark brown ...	155·9	34·6	177·0	95·79	1·00	3·11	91·68	290·0
Iceland train ...	red	160·8	46·4	181·0	95·84	1·68	0·63	93·53	289·4
Newfoundland train	pale yellow ...	149·0	1·4	195·9	96·83	0·29	0·29	96·25	285·3
Seal oil	reddish-yellow	146·2	43·1	184·4	95·96	0·79	0·75	94·42	286·7
Whale oil ...	reddish-yellow	116·2	49·5	184·9	96·70	0·69	0·57	95·44	289·0
,, ...	pale yellow ...	106·1	4·2	177·3	95·19	0·70	1·44	94·33	293·9
,, ...	reddish-yellow	105·8	51·4	176·7	96·49	1·77	0·39	94·33	298·9

F. H. L.

Valuation of Resin. A. Heupel. (*Zeit. angew. Chem.*, 1899, 171.)—The results of more than 1,000 determinations of the acid and saponification numbers of colophony gave an average of 100 for the acid number and 170 for the saponification number, and a similar difference was found between these numbers with other resins. The author agrees with Schick that the determination of constants as a basis for the valuation of colophony is useless. Out of many thousands of casks of resin which he has examined, he has never found two to give the same constants.

H. H. B. S.

Contributions to our Knowledge of Colophony. R. Schick. (*Zeit. angew. Chem.*, 1899, 172.)—The author replies to Dieterich's remarks in the *Zeit. angew. Chem.*, No. 5. He again points out that Dieterich's "acid number," obtained by allowing the resin to stand for two hours in the cold, with excess of alcoholic potash and titrating back can be neither an acid number nor a saponification number, since it is greater than the number obtained by direct titration, and less than that obtained on standing for twelve hours in the cold. He considers that the sharpness of the end-reaction and the regularity of the decomposition which takes place on direct titration point to the correctness of this method of determining the acid number.

Among other reasons for objecting to the direct method of titration, Dieterich states that this method gives numbers below the calculated acid number of abietic

anhydride. The author replies to this by referring to the numbers given by Dieterich himself (155·85 to 170·80 and 168·00 to 183·50), and by pointing out that Perrenoud has already shown (*Chem. Zeit.*, 1885) that colophony does not consist solely of abietic anhydride. Further, Dieterich himself asserts in the same paper that the resins (and therefore presumably colophony) are to be regarded as mixtures of very uncertain composition.

The author does not attach any importance to the iodine number as a means of valuation, the fluctuations (as stated by Dieterich) being too great (*cf.* ANALYST, this vol., 77). H. H. B. S.

A Reaction of Gallic Acid, Tannin, and Pyrogallol. G. Griggi. (*Boll. Chim. farm.*, 1899, xxxviii., 5; through *Chem. Zeit. Rep.*, 1899, 29.)—When a few c.c. of a 1 per cent. solution of gallic acid are shaken with 1 c.c. of a 3·3 per cent. solution of potassium cyanide, a bright ruby-red colour is produced, which disappears on standing, but is re-formed every time the tube is agitated in presence of air. A drop or two of hydrogen peroxide also colours the bleached liquid similarly. A 1 per cent. solution of tannin or pyrogallol gives a yellowish-red colour with potassium cyanide, and the subsequent decolorization is slower. Agitation and hydrogen peroxide reproduce the tint, but an excess of the latter gives a permanent yellowish-brown colour with gallic acid, and a characteristic dirty-white precipitate with tannin. F. H. L.

A New Method of Estimating Tannin. L. Vignon. (*Bull. Soc. Chim.*, 1898, xix., 923-926.)—This is based on the fact that ungummed silk can absorb tannin quantitatively from an aqueous solution, leaving the other substances which usually accompany it (gallic acid, glucose, etc.). The silk is used in large excess of the tannin, which should be in dilute solution, the proportion being about 5 grammes of silk to 0·1 gramme of tannin in 100 c.c. of water. The tannin is absorbed after four or five hours at 50° C., and can then be determined by any of the usual methods; titration of the liquid with permanganate, using indigo as indicator, gave the most exact results.

The silk (ungummed) is prepared by being washed and rinsed in distilled water and dried, and 5 grammes are used for each estimation.

Absorption by silk has the advantage over absorption by violin-strings or hide-powder of being much more rapid, while, unlike hide-powder, it does not yield soluble organic matter to the solution. Finally, the colour of the silk after the absorption enables one to estimate comparatively the degree of coloration of the tannin—an indication which is of value to the dyer and to the tanner. C. A. M.

INORGANIC ANALYSIS.

Gravimetric Estimation of Sulphuric Acid in presence of Iron. The Importance of the Ionic Theory in Analytical Chemistry. F. W. Küster and A. Thiel. (*Zeits. anorg. Chem.*, 1899, xix., 97.)—It is now well known that sulphuric acid cannot safely be thrown down by means of barium chloride from a solution con-

taining dissolved iron; for inasmuch as the impurity in the precipitate takes the form of ferric sulphate, which is decomposed into oxide on ignition, part of the acid is lost, and attempts to purify the barium sulphate by extraction of the iron simply lead to increased deficiency in the yield, because they remove that portion of ferric oxide which, left behind, would partially take the place of the weight of sulphuric anhydride that was not originally combined with barium. The usual method of precipitating the iron first with ammonia and treating the *filtrate* with barium chloride consumes much time, and is also inaccurate, since some of the acid is carried down with the ferric hydrate as a basic sulphate. The authors find, however, that when barium chloride is added to a liquid in the presence of iron, the precipitate falls with absolute purity provided the iron is either (1) in an insoluble form, *i.e.*, a state of suspension; or (2) is in solution combined with a complex ion, *e.g.*, tartaric, or preferably oxalic acid.

A solution of sulphuric acid and ferric chloride containing 2 atoms of iron to 3 of sulphur (the proportion occurring in pyrites) was prepared of such a strength that 25 c.c. should theoretically yield 0·713 gramme of $BaSO_4$. This volume was diluted with an equal amount of cold water, and an excess of ammonia (20 c.c.) introduced. The whole was heated almost to the boiling-point with constant stirring, and, neglecting the ferric hydrate, 15 c.c. of about 10 per cent. barium chloride solution were dropped in slowly. Eight c.c. of 10 per cent. hydrochloric acid were next added, and the liquid kept warm and well mixed for two hours. It was then set aside to cool for half an hour, the liquid run off through a filter, and the residue treated several times in succession with 2 c.c. of cold HCl and 30 c.c. of boiling water, allowing each extraction to take place for thirty minutes. Finally the barium sulphate was washed three times by decantation with boiling water, brought on to the filter, washed, dried, and weighed. The precipitate was perfectly white, and weighed from 0·7109 to 0·7142 (mean 0·7125) gramme: 0·5 milligramme, or 0·07 per cent., less than that required by theory. In the second process, 25 c.c. of the acid ferric chloride liquor were mixed with 15 c.c. of water and 25 c.c. of a cold saturated solution of ammonium oxalate, heated to the boil, precipitated with 15 c.c. of barium chloride, and acidified with 15 c.c. of HCl. The further treatment was precisely as above: the yield was almost pure white, and weighed 0·7115 or 0·7129 (mean 0·7122) gramme, *i.e.*, 0·8 milligramme, or 0·11 per cent., less than demanded by theory. A tartaric acid solution gave very similar results, and the barium sulphate was ultimately of perfect purity; but the employment of this acid is not to be recommended, for much more iron is first carried down, and the individual yields show wider variations among themselves.

In describing their methods and experiments, the authors express some surprise that the idea of converting the iron into some soluble compound other than sulphate before precipitation does not appear to have occurred to the numerous investigators who have endeavoured to overcome the difficulty of obtaining a barium sulphate uncontaminated with this troublesome and exceedingly common impurity. As Jannasch has conclusively shown that the precipitated iron always takes the shape of ferric sulphate, it is clear that such precipitation cannot occur unless the liquid contains ions both of ferric iron and of sulphuric acid. The latter obviously cannot

be avoided; the former may, either by throwing the metal entirely out of solution, or by combining it with some more complex ion which does not behave in an analogous fashion. The effect of precipitating and removing the iron as hydroxide is not perfectly satisfactory, and even with the greatest care must lead to a loss of some 0·5 per cent. of the sulphuric acid.

F. H. L.

Estimation of Boric Acid. F. A. Gooch and L. C. Jones. (*Zeits. anorg. Chem.*, 1898, xix., 417.)—The process described by Thaddéeff (ANALYST, 1898, xxiii., 165) suffers from two defects: there is no proof that a mixture of potassium hydrogen fluoride and potassium borofluoride can be quantitatively separated by extraction with potassium acetate solution; while, as the present authors have definitely ascertained, when such a mixture is evaporated in presence of hydrofluoric acid, some of the boric acid volatilizes. Thaddéeff's method always gives too high a result, presumably because the former source of error is the more serious; obviously it can only be exact if they happen to neutralize one another.

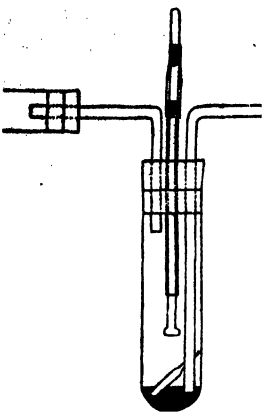
The modified form of Rosenbladt's process, in which a borate is distilled with methylic alcohol and acid into a solution of a known weight of lime, is reasonably accurate. The acid may be nitric or acetic, and should only be employed in sufficient excess to decolorize phenolphthalein, a few drops more being added occasionally during the distillation. In this way the necessity for having such a large quantity of lime, as recommended by Moissan, is avoided. Particularly when nitric acid is adopted, the dried residue of lime and boric acid requires very cautious heating before the actual ignition; even with acetic acid it should be handled carefully to prevent loss. It is perfectly possible to ignite the original calcium oxide to constant weight, and to weigh it without appreciable error in spite of its hygroscopic nature; but to avoid the troublesome manipulation, the authors suggest sodium tungstate as an absorbent.

This salt is fused with a small excess of tungstic acid (to remove traces of carbonate); a known weight of lime (4 to 7 grammes for 0·2 to 0·3 gramme of boric oxide) is dissolved in water, placed in the receiver of the distilling apparatus, surrounded with ice-cold water, well agitated with the boric acid vapours, allowed to rest half an hour, and then evaporated, melted, and weighed. Using acetic acid, the residue must be ignited till it is perfectly colourless on cooling. Either nitric, acetic, or sulphuric acid can be employed—excess of the latter is objectionable. Various examples are quoted; the results are fair, but tend to be a little too low.

F. H. L.

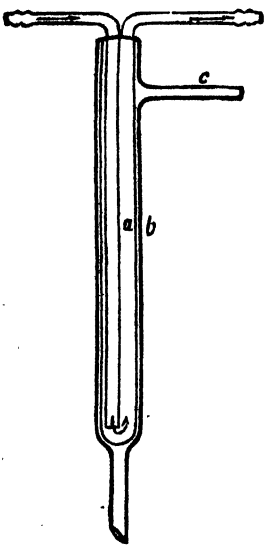
APPARATUS.

Estimation of Carbon and Hydrogen in Volatile Organic Liquids. F. W. Streatfield and L. Eynon. (*Chemical News*, vol. lxxix. [2045], p. 50.)—The apparatus devised by the authors for this purpose consists of a strong, moderately large test-tube containing a small quantity of mercury and fitted with a triple-bore caoutchouc stopper. The tube from the supply of air



or oxygen dips into the mercury, to prevent backward diffusion; a second tube leads to the combustion tube; and a glass rod with a flattened head passes loosely through a tube fixed in the central bore of the stopper, communication with the external air being prevented by a joint of rubber tubing. The substance is introduced into the test-tube, enclosed in a sealed bulb which is broken by means of the rod, and the vapour is carefully expelled into the combustion tube by a current of air or oxygen entering through the supply tube. If the vapours are capable of attacking the caoutchouc fittings, the apparatus can be inverted, the air-supply tube being in such event bent upon itself so as to dip into the mercury seal. C. S.

A Condenser for Extraction Work. C. G. Hopkins. (*Journ. Amer. Chem. Soc.*, vol. xx. [12], pp. 965, 966.)—This condenser obviates the trouble caused by condensed atmospheric moisture collecting on the outer surface of Liebig or Allihn condensers and running down over the extraction apparatus.



It consists of a closed tube *a* of thin glass, 25 cm. long and 25 mm. external diameter, into the top of which are sealed two glass tubes about 6 mm. in diameter, as water inlet and outlet, one of them extending nearly to the bottom of *a*. The whole is enclosed, by sealing, in a stronger glass tube *b*, 30 mm. in internal diameter, drawn out below, for a length of 8 mm., to 10 mm., and ground off obliquely at the end. A lateral tube *c*, 5 cm. long and 12 mm. wide inside, is sealed on to *b*, about 3 cm. from the top, to facilitate connection with a drying tube.

The vapour being distributed in a thin layer over a large cooling surface, greater efficiency is obtained; the risk of breakage, through unequal expansion below and above the water-line, is nil; and the outer surface of the condenser is too warm to condense atmospheric moisture. C. S.

MISCELLANEOUS.

Antiseptics in Food.—A paper on the subject of preservatives or antiseptics in food was read by Dr. Alfred Hill, the Medical Officer of Health and Public Analyst for the City of Birmingham, at a recent meeting of the Incorporated Society of Medical Officers of Health. A discussion ensued, in which a number of medical officers of health and public analysts took part, and ultimately the following resolutions were adopted: (1) That the Incorporated Society of Medical Officers of Health strongly disapproves of the practice of adding preservative chemicals to milk and other foods. (2) That if preservative chemicals are added to any food, a full disclosure as to the nature and amount thereof should be made to the purchaser.

THE ANALYST.

JUNE, 1899.

PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS.

THE monthly meeting of the Society was held on Wednesday evening, May 3, in the Chemical Society's Rooms, Burlington House, the President (Mr. W. W. Fisher, M.A.) occupying the chair.

The minutes of the previous meeting were read and confirmed.

Certificates of proposal for election to membership in favour of the following candidates were read for the second time: Edward Halliwell, F.I.C.; Arthur John Starey; and William Lincolne Sutton, F.I.C.; and in favour of the following candidates for the first time: Thomas Waterworth Glass, B.Sc. (Lond.), Analyst, London; Henry Dixon Hewitt, assistant to Mr. Thomas A. Pooley, B.Sc., London; Daniel Lewis Thomas, Public Analyst for the Limehouse District, London; and Samuel Allinson Woodhead, B.Sc., Public Analyst for East Sussex, Uckfield.

Messrs. Julian L. Baker, F.I.C., Herbert E. Burgess, L. C. Deverell, Julius Lewkowitsch, Ph.D., F.I.C., F. R. O'Shaughnessy, Assoc. R.C.S., Arthur Marshall, A.I.C., and Edward T. Shelbourn, A.I.C., were elected members of the Society.

The following papers were read: "The Assay of Belladonna, B.P.," by F. C. J. Bird; "On the Use of Boric Acid and Formaldehyde as Milk Preservatives," by S. Rideal, D.Sc., and A. G. R. Foulerton, F.R.C.S., and "The Value of the Estimation of Pentosans in Food Materials," by Otto Hehner and W. P. Skertchly.

NOTE ON BORIC ACID IN MILK SAMPLES.

BY E. G. CLAYTON.

(*Read at the Meeting, March* 1, 1899.)

BORIC ACID was tested for qualitatively in 403 samples of milk received by me for analysis during the years 1893-98, and the following is a record of the results:

Total number of samples, 403; of these 164, or 40·6 per cent., contained boric acid. Thus:

In 1893, of	45	samples,	28	contained boric acid, or 62·2 per cent.
1894, ,,	28	,,	17	60·7
1895, ,,	92	,,	43	46·7
1896, ,,	62	,,	29	46·7
1897, ,,	108	,,	37	34·2
1898, ,,	68	,,	10	14·7
	403		164	

In the table on the opposite page the samples are classified according to quality, and the relative amounts of boric acid added. The numbers of adulterated, doubtful (that is, poor or inferior, or *probably* adulterated), and genuine samples are also shown. Watered milks and samples with the fat abstracted are indicated respectively by the letters w and s; both sophistications together are shown by w/s. The figures demonstrate a gradual diminution in the use of boric acid, possibly owing to the introduction of formaldehyde, which, however, was not tested for in any of these samples.

DISCUSSION.

The PRESIDENT desired to ask by what method the author had tested for boric acid. He himself had latterly found that the most expeditious way was one which had been described several years previously in the ANALYST (xvi., 180), viz., to heat 0·5 c.c. of the milk in a dish with a little weak and slightly acid tincture of turmeric. The reaction was thus obtained more readily than by any other method that he had tried.

Mr. CLAYTON said that he had tested the ash after acidification by hydrochloric acid with turmeric paper, confirming the test by applying alkali to the turmeric paper after drying. The alkali used was generally potash, but sometimes ammonia.

NOTES ON MILK ANALYSIS.

BY L. DE KONINGH, F.I.C.

(*Read at the Meeting, April* 12, 1899.)

Analysis of Sour Milks.—An attempt to analyse sour milks by the specific gravity process has already been successfully made by Weibull (*Chem. Zeit.*, 1895), the process being briefly as follows: The milk is mixed with a definite volume of ammonia of known specific gravity to the extent of about 25 per cent., the whole is well shaken until the milk solids have redissolved, and the volume is then carefully measured, the exact quantity of milk used thus being found; the specific gravity of the mixture is taken, and that of the milk found by calculation.

The process, I am informed, is accurate, but the result greatly depends on the skill of the operator; it also renders the estimation of the fat by the Leffmann-Beam process less accurate. The process which I now wish to bring to your notice is much more simple and less dependent on analytical skill, and, therefore, more suited for a commercial laboratory.

The whole, or at least the greater portion, of the well-mixed sample is emptied into a measure; an improvised one, having a mark 95 c.c., and also two other marks, 100 and 105 c.c. respectively, will generally be found to answer the purpose. After pouring in the milk up to the mark 95, solution of caustic soda is added up to 100 c.c., the whole is emptied into a dry flask and gently shaken for five minutes. The solution of the soda is made by partially filling a large cylinder with water at nearly 60° F., and then gradually adding strong aqueous soda until the specific gravity is exactly 1,030 (water = 1000) at 60° F. This gravity being the same as that of

Year.	Samples Containing $B(OH)_3$.	Samples Free from $B(OH)_3$.	Nature or Quality of Sample.	$B(OH)_3$ Reaction Observed.				Totals.
				Considerable.	Moderate.	Trace.	None.	
1893	28 samples { 16 adulterated; 4 doubtful; 8 genuine	17 samples { 8 adulterated; 4 doubtful; 5 genuine	Adulterated	(2) w s	(11) w w w w w w w w s s s	(3) s s s	(8) w w w w w w w s	24
			Doubtful	—	3	1	4	8
			Genuine	—	6	2	5	13
			Total ...	2	20	6	17	45
1894	17 samples { 12 adulterated; — doubtful; 5 genuine	11 samples { 4 adulterated; 1 doubtful; 6 genuine	Adulterated	(3) w/s s s	(9) w s s s s s s s s	—	(4) w w s s	16
			Doubtful	—	—	—	1	1
			Genuine	3	1	1	6	11
			Total ...	6	10	1	11	28
1895	43 samples { 9 adulterated; 10 doubtful; 24 genuine	49 samples { 5 adulterated; 11 doubtful; 33 genuine	Adulterated	(4) w/s w w s	(3) s s s	(2) w s	(5) w s s s s	14
			Doubtful	2	6	2	11	21
			Genuine	8	10	6	33	57
			Total ...	14	19	10	49	92
1896	29 samples { 2 adulterated; 5 doubtful; 22 genuine	33 samples { 5 adulterated; 2 doubtful; 26 genuine	Adulterated	—	—	(2) w s	(5) w w/s w/s s w	7
			Doubtful	—	4	1	2	7
			Genuine	1	11	10	26	48
			Total ...	1	15	13	33	62
1897	37 samples { 5 adulterated; 5 doubtful; 27 genuine	71 samples { 17 adulterated; 9 doubtful; 45 genuine	Adulterated	—	(3) w/s w/s s	(2) w/s w	(17) w/s w/s w/s w/s w/s w w w w s s s s s s s s	22
			Doubtful	1	2	2	9	14
			Genuine	11	2	14	45	72
			Total ...	12	7	18	71	108
1898	10 samples { — adulterated; 1 doubtful; 9 genuine	58 samples { 5 adulterated; 9 doubtful; 44 genuine	Adulterated	—	—	—	(5) w w w s s	5
			Doubtful	—	—	1	9	10
			Genuine	4	1	4	44	53
			Total ...	4	1	5	58	68

average milk, a c.c. more or less of the alkali does not affect the experiment. The alkaline milk is now emptied into the ordinary test-glass, and its specific gravity taken at 60° F. with a delicate milk-hydrometer; if the temperature should differ from 60° F., then correct by means of Richmond's sliding rule. If the action of the soda has not been successful, empty the milk into the measure and add another 5 c.c. of soda; in one obstinate case 15 c.c. was required, but if the sample is not too stale 5 c.c. will always be sufficient. The specific gravity found may be assumed to be correct if the mixture has a density not greatly varying from 30; but if this is much higher or lower, the following correction should be applied: If 5 c.c. of alkali have been used, then deduct from the gravity found 1·5 and divide the difference by 0·95; if 10 or 15 c.c. have been used, substitute 3 or 4·5 for 1·5 and divide by 0·90 or 0·85 respectively.

When trying the accuracy of the process with samples, the specific gravity and fat of which were carefully taken when quite fresh, it was found that the specific gravities as found by the soda process were rather lower than might be accounted for by decomposition of the samples. The remarkable fact was now disclosed that when treating fresh milk with from 5 to 15 per cent. of the soda solution the gravity is instantly lowered, the hydrometer moving not quite so fast, although very regularly. I am at a loss to account for this phenomenon, but think it not improbable that it may be, partially at least, caused by the precipitation of the calcium triphosphate. The loss in specific gravity is, however, as far as my experiments go, fairly constant, and I therefore propose adding 0·8° in order to get the true gravity of the sour sample, and fancy that the result will be the same as that of the original milk minus the loss in specific gravity caused by decomposition.

To prevent disappointment, I wish to state particularly that I have no idea how the process will work with samples which are in an advanced state of decomposition, and deserve the name of "putrid." But I think the process will be found useful to those analysts who get their samples from a distance, or who are unavoidably prevented from touching them the very day they are brought in, and then find them unsuitable for the gravity process next morning.

The alkaline liquid may now be advantageously used for the estimation of the fat by the invaluable Leffmann-Beam process. If 5 c.c. of soda solution have been used, then add an extra c.c. to save the trouble of correcting for dilution.

Detection of Cane-Sugar.—It cannot be denied that the ordinary non-fatty-solids process is not alone conclusive, and that it is a very easy matter for intelligent milk-vendors to defeat the authorities by adding to their milk a solution of sugar, or highly dilute sweetened condensed milk. This, if much overdone, may be readily detected by the taste, but I quite agree with an experienced private inspector, who himself carried out some experiments, that although one may swear to the presence of 15 per cent. of sugar-water of 8 per cent. strength (this sugar solution has a density of 1030·8, and consequently defeats the gravity test), less than 10 per cent. is likely to be overlooked. Until recently it was not possible to certify the presence of sugar without making a regular chemical analysis coupled with polarimetric observations, and if the proportion of cane-sugar is below 1 per cent., I am somewhat doubtful as to the results so obtained. Moreover, the process is too complicated and takes too much time to be

of any use, either in a public or private laboratory, where the analyst is often required to give in his result the same day. Fortunately a test has recently been published (ANALYST, 1898, 37) which is based on the reduction of molybdic acid by cane-sugar, a fact already used for detecting simple syrup in commercial glycerin. Cotton, noticing that lactose has no reducing action, proposed the following test: 0·5 gramme of powdered ammonium molybdate is dissolved in 10 c.c. of milk, 10 c.c. of dilute hydrochloric acid (1 : 10) is added and the mixture heated slowly to 80° C., when the presence of cane-sugar will betray itself by a blue coloration, even when its amount does not exceed a few tenths of a per cent.; pure milk is said to be unaffected.

I have tried the process, and it certainly will detect 0·4 per cent. of cane-sugar. In practice I find it more convenient to keep a saturated solution of the reagent ready for use in a narrow graduated measure, 2 c.c. sufficing for one test; 8 c.c. of acid (1 : 8) is then added. The mixture first turns yellow owing to the formation of phospho-molybdate, but this precipitates along with the curd. The test-tube is now placed in water, which is then gradually heated to 80° C., and then kept for five minutes at that temperature, which should not on any account be exceeded. Those practising the process for the first time should make a blank experiment side by side with pure milk, so as to accustom the eyes to the slight but differently shaded colour always given by a genuine sample. With a little practice the process will be found to be a valuable one, and will enable the operator to state within fifteen minutes whether there is sufficient cane-sugar to unduly increase the non-fatty solids; the approximate amount may be judged by trying side-by-side a sample purposely mixed with, say, 10 per cent. of sugar solution. I tried whether decomposed milk gives any reduction, but find this is not the case; on the other hand, milk containing 5 per cent. of sugar-water and kept for fourteen days only gave a very unsatisfactory test, but another sample containing 10 per cent. still gave a decided reaction. It was also tried whether the presence of "colouring" might deceive the analyst. Annatto, the dye almost universally used in London, when added to milk in such quantity as to render the article unsaleable, does give a slight reduction, but the amount used in practice is so small that it does not interfere with the test.

Detection of Boric Acid.—It is well known that the incineration of a large quantity of fatty organic matter causes a loss of acid; the rapid evaporation of a large quantity of milk and the burning of its residue is also a very unpleasant operation. A good deal of inconvenience is avoided by adopting the following plan: 100 c.c. of milk are put into a large beaker covered with a light basin containing some cold water. The milk is now rapidly heated to boiling, the flame is at once removed, and 8 c.c. of very weak nitric acid (1 in 50) are poured in from a small test-tube. When cold the liquid is filtered, and an actual experiment has proved that this volume of acid just compensates for the volume occupied by the curd from good average milk; when dealing with separated milk 6 c.c. are sufficient. As there is no need to have an absolutely clear filtrate, the milk may be rapidly filtered through muslin and 80 c.c. collected. One eighth of a gramme of sodium carbonate is now added, and the whole is rapidly boiled down in a spacious platinum dish and finally burnt to a gray ash; the nitrate greatly assists in the burning, without, however, causing the least deflagration. The water-soluble portion of the ash is then tested for

boric acid either qualitatively or quantitatively by one of the now numerous methods. If done by the fluoride process it is preferable to use potassium instead of sodium carbonate. I have found that the aqueous solution of the ash can be obtained practically free from phosphoric acid in the following manner: Before adding the soda, add to the hot solution 2 drops of a saturated solution of calcium chloride and then a little ammonia. When cold filter off the calcium phosphate, which, however, in this case is not precipitated so completely as might be expected, distinct traces being afterwards found in the insoluble ash. As the filtration of the calcium phosphate is often a little slow, the filter-pump should be used.

NOTE ON A POSSIBLE SOURCE OF ERROR IN MODIFICATIONS OF THE LEFFMANN-BEAM METHOD FOR ESTIMATION OF FAT IN MILK.

By H. Droop Richmond and F. R. O'Shaughnessy.

(*Read at the Meeting, March* 1, 1899.)

Leffmann and Beam recommended the use of amyl alcohol mixed with an equal bulk of hydrochloric acid for their centrifugal method of fat estimation; one effect of the mixture was that the amyl alcohol was well distributed throughout the milk by the curdling effect of the hydrochloric acid before the sulphuric acid was added.

The modification of Gerber, in which the hydrochloric acid is omitted, has come into general use; his instructions were to add first the sulphuric acid, next the amyl alcohol, and finally the milk. He drew attention to the fact that the sulphuric acid and amyl alcohol could not be left long in contact without vitiating the results, and as an alternative recommended that the acid be added first, next the milk, and finally the amyl alcohol.

During the summer of 1897, it was found that when the acid and amyl alcohol were accidentally shaken up together (Gerber's original instructions being followed) before adding the milk, the fat obtained on centrifugalizing was frequently highly coloured, and the resulting estimation was invariably higher than when the acid and amyl-alcohol were not allowed to mix. The difference sometimes reached 0·5 per cent. We found that this effect was much more pronounced on very hot days, and in cold weather practically disappeared.

The following experiments will show that when amyl alcohol and strong sulphuric acid are mixed an action takes place resulting in the formation of a liquid compound which is insoluble in a more dilute acid, while a mixture of sulphuric acid, water, and amyl alcohol results in the formation of no such compound. The action between sulphuric acid and amyl alcohol increases at higher temperatures:

I. A milk giving 3·63 per cent. of fat by gravimetric estimation was taken and examined in the following way. The reagents were added in the following order:

a.	Sulphuric acid	10 c.c.	*b.*	Sulphuric acid	10 c.c.	*c.*	Amyl alcohol	1 c.c.
	Milk	11 ,,		Amyl alcohol	1 ,,		Sulphuric acid	10 ,,
	Amyl alcohol	1 ,,		Milk	11 ,,		Milk	11 ,,

One series of experiments was made at 14° C., and another at 29° C., which represent the temperatures of a laboratory in winter and summer.

The following mean results were obtained:

Temperature	...	...	14° C.	...	...	...	29° C.
a.	...	...	3·66 per cent. fat	...	...		3·63 per cent. fat.
b.	...	...	3·67 ,,	...	...		3·65 ,,
c.	...	...	3·76 ,,	...	...		4·11 ,,

The fat of *c* at 29° C. was darker than the others, and an odour was noted in this, but not in the others, which resembled that obtained on warming sulphuric acid and amyl alcohol. This experiment was several times repeated, with similar results.

II. To exaggerate the action and to see the effect of temperature, mixtures were made containing:

a.	Sulphuric acid	10 c.c.	*b.*	Sulphuric acid	15 c.c.
	Amyl alcohol	1 ,,		Amyl alcohol	1·5 ,,

One series was cooled during mixture and kept at 14° C. for fourteen hours, and the other series was not cooled and was left at 37° C. for fourteen hours; water was then added to make up to 30 c.c., and the separated layer measured. The results were:

Temperature	...	...	...	14° C.	...	...	...	37° C.
a.	...	...	...	0·295 c.c.	...	...	...	0·590 c.c.
b.	...	...	...	0·424 ,,	...	...	...	0·800 ,,

The layers at 14° C. were nearly transparent, while at 37° C. they were deeply coloured.

III. Ten c.c. sulphuric acid, 11 c.c. of water, and 1 c.c. amyl alcohol (all at 29° C.) were mixed; no separation occurred in centrifugalizing, and the solution only became light brown. One c.c. amyl alcohol, 10 c.c. sulphuric acid, and 11 c.c. water (all at 29° C.) were mixed and centrifugalized, a liquid layer equivalent to about 0·2 per cent. fat was obtained, and the liquid became violet.

IV. A series of experiments were made on the rate of heating of sulphuric acid, amyl alcohol, and mixtures of these two bodies in various proportions. The liquid to be heated was placed in a tube, contained in a second tube, which latter was immersed in boiling water. The difference in temperature between the liquid and water was taken, and the logarithms of a series of numbers so obtained were plotted against their corresponding times. The resulting curves, in the case of amyl alcohol which had been previously heated and of sulphuric acid, were straight lines. In the case of the mixtures in various proportions of these two bodies the curves all showed an increasing rate of rise, indicating that continued chemical change was taking place. Amyl alcohol which had not been previously heated showed an abnormal rate of rise, indicating a change on heating which is not continuous.

From our experiments we conclude:

1. To obtain correct results with the Gerber method it is advisable to always employ the alternative method, *i.e.*, to add first sulphuric acid, next milk, and finally amyl alcohol.

2. The modification of the method according to which amyl alcohol is added

first has a tendency to give high results, and at high temperatures (in hot weather) the error may assume serious proportions.

3. Gerber's original method gives good results in the hands of a careful operator, but may at times give rise to error.

We would point out that the order of adding reagents does not appear to be considered an essential part of the method, and there is no reason against Gerber's alternative order of adding reagents being always followed.

DISCUSSION.

The PRESIDENT having invited discussion,

Dr. DYER said that when he first used the Gerber apparatus, working by the original directions, he had often found charring to occur when the milk was put into the acid. When the milk was put in first, the fusel oil next, and the acid last, good results were obtained.

Mr. O'SHAUGHNESSY said that, according to their experience, it was more difficult to make a satisfactory mixture when the milk was put in first, owing to the curdling of the milk by the initial action of the acid.

ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

FOODS AND DRUGS ANALYSIS.

The Influence of Distillery Refuse on Cow's Milk. A. Petermann. (*Bull. de l'Ass. belge*, 1899, xiii., 148.)—The author has examined samples of milk taken at intervals of three months from a dairy of fifteen cows fed on maize wash from a distillery in Namur, but no trace of alcohol could be detected in 1 litre of milk.

The author's experience in this respect differs from that of Weller (ANALYST, xxii., 264). C. A. M.

Proportion of Water in Butter. B. Martiny. (*Landw. Jahrb.*, 1898, xxvii., 773; through *Chem. Zeit. Rep.*, 1899, 46.) As the result of more than 20,000 separate tests of different specimens of butter, the author finds the following average figures:

Country.	No. of Samples examined.	Water Per cent.
Germany, fresh	523	13·96
,, salt	1,107	13·01
Denmark	9,847	13·99
Sweden	4,423	13·66
Finland	438	11·18
England	334	12·08
France	225	13·40

F. H. L.

The Alteration of Milk-Fat in the Ripening of Cheese. H. Weigmann. (*Zeit. Untersuch. Nahr. Genussm.*, 1899, ii., 31, 32.)—Referring to Kirsten's recent communication (ANALYST, xxiv., 34), the author states that in his opinion the evidence there brought forward is insufficient to disprove the conclusions arrived at by Backe and himself (*Landw. Vers. Stat.*, 1898, li., 1-14), and also by Windisch and by Duclaux. He considers that all that Kirsten has proved is that the fat obtained by Devarda's method from the ripe cheese has the same composition as the fat from the same cheese before ripening. But, inasmuch as the treatment with potassium hydroxide solution removes all the free fatty acids as well as the decomposition products of the milk-sugar, he contends that Kirsten's experiments only show that Devarda's process is an excellent method for extracting from the cheese that part of the fat which has not undergone any change. C. A. M.

Detection of Cotton-Oil Stearin ("Vegetal") in Lard by means of the Phytosterin Test. Notes on the Bömer Process. F. Wirthle. (*Chem. Zeit.*, 1899, xxiii., 250.)—It has recently been suggested in the *Chem. Zeit. Rep.* that a certain proportion of cotton-oil stearin might be present in American lard without being capable of detection by Bömer's phytosterin test (*cf.*, however, ANALYST, 1898, xxiii., 283). The author has accordingly examined a lard adulterated with 10 per cent. of "vegetal," and has obtained the kind of crystals characteristic of a mixture of cholesterin with phytosterin (*i.e.*, short, compact, telescope-like needles).

As regards Bömer's process, Wirthle considers the Kreis and Wolf modification (ANALYST, 1898, xxiii., 294) more complicated than the original, and prefers to work as Bömer prescribed, using, however, only 25 grammes of fat. It is also advisable to add a larger volume of water to the alcoholic soap solution, viz., 130 instead of the 100 c.c. ($=\frac{200}{2}$), recommended by Bömer. The separation may be carried out successfully, and the cholesterin and phytosterin recovered pure, even from rancid fats, if the alcoholic residue after the second saponification is treated with animal charcoal before being crystallized from absolute alcohol; but from utterly putrid samples, some years old, no crystalline matter could be obtained.

"Vegetal" is a yellow substance, semi-solid at ordinary temperatures, which remains liquid a long time after it has once been melted. Its iodine number is 89·24, its saponification number 196·1, and its refractometer number, at 25° C., 63·3. It gives the Becchi and Welmans reactions powerfully. "Vegetal" is not pure cotton-oil stearin, but is partly composed thereof. F. H. L.

Estimation of Sugar in Molasses. O. Foerster. (*Chem. Zeit.*, 1899, xxiii., 196.)—Processes for the determination of sugar in molasses which are based on dissolving the substance in water and making up the liquid to a definite volume are inaccurate, owing to the uncertainty respecting the amount and composition of the insoluble residue; it is better, therefore, to recover the sugar by means of absolute alcohol. A multiple (n) of 13·024 grammes of the sample is rubbed down with spirit, placed in a Sickel extractor over a fine platinum gauze, and allowed to extract for

twelve hours. The spirit is driven off, the mass diluted to n times 75 c.c. with water, 75 c.c. are inverted, 50 c.c. are made up to half a litre, and 25 c.c. of the product (equal to 0·3256 gramme of molasses) are treated with copper, halving the figures given in Herzfeld's tables. As an alternative, the sugar may be determined optically.

F. H. L.

The Composition of Banana Flour. **A. Petermann.** (*Bull. de l'Ass. belge*, 1899, xiii., 147-48.)—Flour obtained from the banana is widely used in Central America. The starch which is extracted from it is known in Europe as "arrowroot of British Guiana." The starch granules are large and ovoid, and of characteristic form.

The author has made a chemical examination of the flour prepared by himself from the fruit of the *Musa paradisiaca* with the following results:

	Per cent.
Water	5·60
Fatty substances	1·73
Albuminous substances	3·13
Carbohydrates, etc.	82·39
Cellulose	1·22
Mineral matter	5·93
	100·00
Glucose	7·19
Dextrins	3·34
Starch	45·76

C. A. M.

The Detection of Maize Starch in Wheat Meal. **K. Baumann.** (*Zeit. f. Unters. Nahr. u. Genussm.*, 1899, ii., 27-29.)—It is claimed that as little as 1 or 2 per cent. of maize can be detected in wheat flour by the following method: About 0·1 gramme of the meal under examination is mixed with 10 c.c. of a 1·8 per cent. solution of potassium hydroxide, and the test-tube is shaken at intervals during two minutes. Four or five drops of hydrochloric acid (strength about 25 per cent.) are then added, and the tube is again shaken. The liquid must still remain alkaline, since the proteid substances which are precipitated in an acid solution interfere with the regular distribution of the liquid for microscopical examination. When a drop is placed on a glass slip and examined under the microscope, the wheat starch granules will be found completely ruptured, while the maize starch remains unaltered.

For a quantitative estimation, mixtures are prepared containing known quantities of the two starches, which are treated with the reagents, and drops of the liquid are compared with those of equal size from the sample in question. This method is equally applicable to the detection of maize starch in rye meal, the starch granules of which are ruptured by the potassium hydroxide even more rapidly than wheat starch. The advantages claimed for potassium hydroxide as a reagent over chloral hydrate are that the disintegrating action can be arrested at any given moment by the addition of acid, and that the wheat or rye starch granules are completely

ruptured without the maize being attacked, whereas with chloral hydrate a considerable proportion of the maize granules are burst, while a third or a quarter of the wheat starch still remains intact. C. A. M.

A Contribution to our Knowledge of Wine Vinegar. K. Farnsteiner. (*Zeit. für Untersuch. der Nahr. und Genussmittel*, 1899, 198.)—The author has studied the changes which take place during the acetous fermentation of wine. Three different kinds of wine were experimented with, and the principal results obtained embodied in the following tables:

Grammes in 100 c.c.	I.			II.			III.		
	30-1-'98	23-3-'98	31-5-'98	30-1-'98	23-3-'98	31-5-'98	30-1-'98	23-3-'98	31-5-'98
Specific gravity at 15° C.	0·9987	1·0036	1·0055	1·0095	1·0173	1·0262	1·0055	1·0112	1·017
Alcohol	5·95	4·29	3·75	4·23	1·67	0·0	4·98	2·61	1·23
Extract	1·88	1·82	2·03	3·60	3·44	3·64	2·87	2·40	2·56
Sugar	0·11	—	0·11	0·65	0·73	0·85	0·27	—	0·30
Total acid (acetic)...	1·68	3·33	3·56	1·96	4·92	7·60	1·63	4·14	6·00
Non-volatile acid as tartaric ...	0·33	0·24	0·23	0·40	0·18	0·26	0·39	0·12	0·14
Total tartaric acid...	0·16	0·19	0·19	0·22	0·26	0·26	0·18	0·21	0·20
Glycerin	0·50	0·56	0·72	0·49	—	0·59	0·35	0·34	0·52
Mineral matter ...	0·23	0·26	0·28	0·26	0·27	0·30	0·28	0·30	0·34
Alkalinity of the ash in c.c. of normal alkali	1·60	—	1·70	2·33	—	2·90	2·60	—	2·85
Polarization in 200 m.m. tube ...	±0	+0° 2′	+0° 19′	−0° 48′	−0° 62′	−0° 39′	−0° 21′	−0° 24′	+ 0° 7′

In the course of the investigation the author discovered the presence of a body of an aldehydic nature, which does not appear to be acetaldehyde. H. H. B. S.

Detection of Formaldehyde in Food Stuffs. F. Jean. (*Rev. Chim. Ind.*, 1899, x., 33; through *Chem. Zeit. Rep.*, 1899, 63.)—As formaldehyde yields insoluble compounds with albuminous substances, and as the methods hitherto advocated for its recognition also show other aldehydes, formalin is not easily detected in articles of food. The following process is recommended: 100 c.c. of the sample (milk, for instance) are heated to 70° C. with 4 or 5 drops of sulphuric acid, dry powdered sodium sulphate is added, the mass is distilled, and the first 50 c.c. of the distillate examined. In presence of formaldehyde this gives (1) a red colour with magenta, bleached by sulphurous acid, and turned violet or reddish by hydrochloric acid; (2) a turbidity with aniline water; (3) a yellow precipitate with Nessler's reagent, changing to red or brown on standing; (4) a turbidity with phenylhydrazine hydrochloride, the solution becoming blue on addition of sodium nitroprusside and sodium hydroxide. Conclusive evidence may be obtained by the blue colour produced with dimethylaniline, according to Trillat's suggestion. F. H. L.

Detection of Saccharin in Articles of Food and Drink. A. Hasterlik. (*Chem. Zeit.*, 1899, xxiii., 267.)—In Spaeth's process 50 c.c. of beer are evaporated with a few crystals of copper nitrate to remove hop-bitter principles, mixed with coarse, clean sand and phosphoric acid, extracted with ether and petroleum spirit, the liquid dried, and the residue dissolved in a little weak sodium carbonate. This solution is then tested in various ways. The sweet taste is very characteristic, and will detect 0·001 per cent. of saccharin; but if many samples have to be tried in succession, the tongue becomes fatigued with the secondary bitter taste of the material, and assistance should be obtained in judging the flavour. The alkaline solution may also be evaporated, the solid matter gradually introduced into fused potassium nitrate, the melt extracted with water, acidified with hydrochloric acid, concentrated if necessary, and sulphuric acid sought for with barium chloride: this process serves equally well for the quantitative estimation of saccharin. In the absence of salicylic acid and tannin, the ether residue may be fused for half an hour at 220° or 250° C. with sodium (not potassium) hydroxide, and the resulting salicylic acid tested for in the acidified aqueous solution with ferric chloride. As Abraham has quite recently shown, however (*Rev. intern. falsif.*, 1899, xii., 17), the iron is apt to give doubtful indications, and especially in the case of pastry, liqueurs, etc., the reaction must be received with caution. Börnstein's fluoresceïn test (*Ber.*, 1888, xxi., 3,396) with resorcinol and sulphuric acid is not only useless for beer, as was pointed out by Gantter, but is utterly without value in the examination of any fermented liquid. [The original article includes a table showing the results of this test as applied to various beers, wines, lemonades, syrups, etc., from which it appears that a green (sometimes a red) fluorescence is produced in the majority of instances, even when the ethereal extract has a sour or indifferent taste and when the salicylic acid reaction is absent.] F. H. L.

Solanine in Potatoes, and a Sensitive Reaction for its Detection. Bauer. (*Zeit. angew. Chem.*, 1899, 99.)—The author examined potatoes for solanine, according to the directions of G. Meyer (*Archiv. für experiment. Pathol. und Pharmacol.*, xxiii., 119, 361). The results were as follows:

Potatoes grown in 1897	...	...	0·02 gramme per kilo.
,, ,, 1898	...	...	0·026 ,, ,,

A solution of telluric acid in moderately dilute sulphuric acid is a very reliable reagent for the detection of minute quantities of solanine, producing an intense red coloration on warming. The reaction does not take place with atropine, morphine, quinine, and other alkaloids. H. H. B. S.

Gomberg's Method for Estimating Caffeine compared with other Methods. E. F. Ladd. (*Amer. Chem. Journ.*, vol. xx. [10], pp. 866-869.)—For this the following three solutions are required: The first contains 18 grammes of potassium iodide and 12·65 grammes of iodine per litre, the second 24·8 grammes of sodium thiosulphate per litre, and the third 4·913 grammes of potassium chromate per litre). The thiosulphate solution is standardized against the chromate solution, and the iodine potassium

iodide against the thiosulphate. A weighed quantity of tea, coffee, or other substance, is boiled with water, made up to a known volume, and filtered. An aliquot part of this is treated with basic lead acetate, until a precipitate ceases to form. After settling, the precipitate is filtered off, and the lead removed from the filtrate with hydrogen sulphide. The liquid, after being boiled to remove dissolved gas, is divided into two equal parts, one of these being acidulated with either sulphuric or hydrochloric acid. To both of these are added a definite quantity of the iodine potassium iodide solution, and, after settling for ten minutes, each is filtered off and titrated with $\frac{N}{10}$ thiosulphate solution. The acidulated portion serves to show if there is any substance present capable of being precipitated by Wagner's reagent; if there is, the corresponding correction is made. One c.c. of $\frac{N}{10}$ sodium thiosulphate solution corresponds to 0·00485 caffeine.

In the case of Vité's method, when working on pure caffeine, there was a loss of 10 per cent., and with tea the results came out as low as 42 per cent. below those yielded by Gomberg's method. Apparently the caffetannate in tea and coffee is not wholly broken up by the lead oxide.

Gomberg's method gave the best results, 99·9 and 100·5 per cent. of caffeine being recovered when working with 0·1 and 0·2 gramme respectively of the pure substance; and higher values than yielded by the other methods were obtained from teas.

For pure caffeine, Peligot's method proved about equal to Gomberg's, but with teas a loss of caffeine results from the use of an excess of lead and prolonged treatment with hydrogen sulphide.

Crosschoff's method gave good results with pure caffeine, but with teas the values came out from 0·355 to 0·786 per cent. below those yielded by the Gomberg method, which is, moreover, simple, and the most suitable one for rapid work.

C. S.

Estimation of Sinapine. **F. Bek.** (*Farmaz. J.*, 1899, xxi., 118; through *Chem. Zeit. Rep.*, 1899, 99.)—The author finds that by using the double thiocyanate of potassium and platinum (Guareschi's reagent for alkaloids) in place of the simple potassium salt, a higher yield of sinapine is obtained from mustard, and also that *Sinapis nigra* does contain some alkaloid, Huseman's statement to the contrary notwithstanding. In carrying out an analysis, 100 grammes of mustard flour are repeatedly extracted with 96 per cent. alcohol, the solvent is evaporated, the residue taken up in water, filtered from fat, mixed with the double thiocyanate, allowed to rest for a week, and the precipitate washed with ether. Calculated as sinapine thiocyanate, the comparative results of the processes are:

	Potassium Salt.	Guareschi's Double Salt.
Sinapis alba	0·509 per cent. ...	0·738 per cent.
,, ...	0·410 ,, ...	0·888 ,,
Sinapis nigra	— ,, ...	0·198 ,,

F. H. L.

On the Ash of Certain Descriptions of Cinnamon. G. Rupp. (*Zeit. für Untersuch. der Nahr. und Genussmittel*, 1899, 209.)—A new article of commerce has recently been introduced in Germany under the title "cinnamon powder." It is said to be prepared from broken cinnamon. As the ash and sand of this preparation were found to exceed the limits of genuine cinnamon (5 per cent. of ash with 1 per cent. of sand), the author, at the request of the Chamber of Commerce (Karlsruhe), examined reliable specimens of broken cinnamon, as imported from China and Ceylon. The highest proportions of ash and sand found in these samples were:

	Ash.		Sand.
In the Chinese	6·08 per cent.	...	3·20 per cent.
„ „ Ceylon	6·50 „	...	2·48 „

On cleaning the specimens by picking out the larger stone particles and sifting out the sand, the proportions of ash and sand were much reduced.

As a result of the inquiry, the Government of the Grand Duchy of Baden, pending action by the Imperial Government, has fixed the maximum ash and sand permissible at 6 per cent. and 3 per cent. respectively. H. H. B. S.

Estimation of Alcohol in Tinctura Lobeliæ [Ætherea]. E. Dowzard. (*Pharm. Jour.*, 1899, lxii., 170; through *Chem. Zeit. Rep.*, 1899, 66.)—This operation being complicated owing to the presence of ether, the author makes use of the fact that alcohol combines with calcium chloride. Fifty grammes of the tincture are mixed with 25 grammes of anhydrous sodium sulphate, a large excess of calcium chloride is introduced, the whole allowed to rest for ten minutes, and the ether driven off by heating to 40° C.; water is then added, and the spirit determined as usual.

F. H. L.

ORGANIC ANALYSIS.

Solubility of Methane and Ethane in Fuming Sulphuric Acid. R. A. Worstall. (*Journ. Amer. Chem. Soc.*, 1899, xxi., 245.)—In spite of the fact that the insolubility of the saturated hydrocarbons in fuming sulphuric acid forms one of the fundamental principles of ordinary gas analysis, the author finds that both methane and ethane are actually dissolved on standing over the acid for any length of time. One sample of methane prepared from methylic iodide lost, roughly, 4 per cent. of its volume per day during eleven days, and another from natural gas lost about 1·4 per cent. per day in much cooler weather. Ethane dissolved at the daily rate of 4·7 per cent. Further experiments to determine the absorption of each gas in short intervals of time showed that methane lost about 0·1 c.c. per hour, the corresponding figure with ethane being on an average 0·25 c.c. per hour. In technical gas analysis, therefore, assuming the absence of ethane, the gas may be allowed to rest over fuming sulphuric acid for an hour, if necessary, without serious error; but in presence of ethane fifteen minutes is the maximum; and in all cases the time of contact should be shortened as much as possible. F. H. L.

Detection of Acetone in Urine. R. van Melckebeke. (*Ann. Pharm.*, 1899, v., 49; through *Chem. Zeit. Rep.*, 1899, 84.)—The author considers Penzoldt's process the best: in presence of acetone a freshly-prepared solution of orthonitrobenzaldehyde gives a blue colour in a few minutes' time. It is better to apply the test to a distillate rather than to the urine itself. F. H. L.

Constitution of Oil of Orange Flowers (Neroli). Schimmel and Co.'s April Report. (*Chem. Zeit.*, 1899, xxiii., 330.)—Oil of orange flowers contains a nitrogenous constituent which is intimately connected with its peculiar odour, and which can be extracted by acid. On investigation this body has proved to be the methyl ester of anthranilic acid, distilling at 9 mm. pressure between 124° and 125° C., solidifying on standing, and melting at 25·5° C. Its solutions have a blue fluorescence, and by hydrolysis it yields anthranilic acid melting at 144° to 145°. The substance recovered from the natural oil is thus identical in every respect with the methyl ester synthetically prepared from anthranilic acid. Further details are promised.

F. H. L.

The Composition of Oil of Lemon Grass. H. Labbé. (*Bull. Soc. Chim.*), 1899, xxi., 77-79.)—According to the analysis of F. Tiemann (*Bull. Soc. Chim.* xx. [23], 1) this oil contains only one aldehyde (citral), which forms from 84 to 85 per cent. of the total constituents. As the author's experiments have led him to a different conclusion, he has again examined the oil, using the method of separating citral and citronellal previously described by him (ANALYST, this vol., p. 99).

One hundred grammes of the oil were shaken for two or three hours with an equal volume of a freshly-prepared saturated solution of sodium bisulphite, containing a small quantity of ether to facilitate the crystallization. The crystals were dissolved in water, the solution washed with ether, and a concentrated solution of barium chloride added. The precipitate (18 grammes) was washed with water, alcohol, and ether, and treated with alcoholic potassium hydroxide (5 per cent.) which decomposed it. On extraction with ether, about 6 grammes of an oil with the characteristics of citronellal were obtained. Theoretically at least 8·5 grammes of aldehyde should have been obtained by saponification, but the author states that he has not yet been able to recover the citronellal quantitatively from its barium compound.

The aqueous solution which had been treated with the potassium hydroxide yielded a considerable proportion of citral, and a further quantity which had escaped combination in the first treatment was obtained by again treating the original filtrate with bisulphite. The total amount of citral found was 76 to 77 per cent., or about 7 or 8 per cent. lower than that found by Tiemann. The citral obtained by the second treatment with bisulphite was practically free from citronellal, the bisulphite compound of which is more readily formed than that of citral.

The residue of 24 grammes was found to contain about 4 grammes of geraniol, the presence of which was also mentioned as probable by Tiemann; about 4 grammes of a liquid with an odour of citral (B. P. 200-220° C.); 3 to 4 grammes of a liquid

boiling at 170°-180° C., and judged by its odour and properties to be methyl-heptenone; and a residual portion of 8 to 9 grammes not yet investigated.

C. A. M.

The Composition of Essence of Petit-Grain. E. Charabot and L. Pillet. (*Bull. Soc. Chim.*, 1899, xxi., 74-77.)—The essential oil of petit-grain has usually been described as a slightly dextro-rotatory liquid, and the authors have found the oil from Paraguay to have this property and to be less soluble in 80 per cent. alcohol than the various lævo-rotatory specimens distilled by themselves in Cannes (ANALYST, this vol., p. 100).

The various French samples mixed together gave a product with a rotation of −5·34° at 15° C. and containing 61 per cent. of esters.

This oil saponified and distilled under normal pressure yielded the following fractions:

		°C.	Grammes.	Rotation in 100 millimetre tube.
1	before	185	20	− 9·40°
2	between	185-195	31	−12·45
3	,,	195-197	14	−13·00
4		197-200	110	−13·30
5		200-210	55	− 9·40
6		210-220	24	− 6·48
7		220-232	20	+ 0·20
8	residue and loss		36	

After several fractionations the first fraction was reduced to 11 grammes boiling below 185° C. with a rotation of −6·56°, from which the authors concluded that their native essence did not contain any appreciable quantity of limonene. This terpene was found by Tiemann and Semmler among the constituents of petit-grain, and has also been found by the authors in an American essence, the rotation of which was +0·12°, and which after a single fractionation gave a portion distilling below 190° C. with a rotation of +7·40°. They consider that the presence of the larger proportion of limonene explains the difference in character between the French and American oils, and suggest that this is probably due to French oil being obtained by the distillation of the leaves only, whilst in the preparation of American essences both leaves and oranges are employed.

All the fractions distilling below 197° contained linalol in considerable proportion, and the products boiling between 197° and 200° C. consisted of nearly pure linalol. The fraction boiling between 220° and 232° C. was composed almost exclusively of geraniol, which could be isolated by treatment with calcium chloride and converted into citral by oxidation.

The fractions boiling between 200° and 220° C. were mixtures of linalol and geraniol, the fifth fraction containing 70 per cent. of the former and 30 per cent. of the latter, and the sixth fraction about equal proportions of each, as indicated by the respective rotations.

The authors calculate that essence of petit-grain distilled from the leaves

alone contains after saponification 70 to 75 per cent. of lævo-rotatory linalol ($(\alpha)_D = -15{\cdot}72°$), and 15 to 10 per cent. of geraniol.

The residue yielded a solid substance crystallizing from petroleum spirit, and a liquid giving the reactions of sesquiterpenes. C. A. M.

Formation of Furfural from Starch and its Derivatives. F. Sestini. (*L'Orosi*, 1898, xx., 325; through *Chem. Zeit. Rep.*, 1899, 74.)—If 10 or 15 grammes of crust of bread are heated to 110° or 115° C. in an oven, furfural is given off, and may be recognised by the bright red colour produced on a piece of paper moistened with a solution of aniline acetate and exposed to aqueous vapour. Crumb of bread only shows a similar reaction after it has been dried and baked at a temperature of 140° or 160° till it assumes a hazel-nut tint. Distilled in a current of steam, crust yields no furfural, so that the substance does not exist as such in bread. Commercial or acid-free starch gives the same reaction if it is heated to 185° or 200°; and after moistening with sulphuric or hydrochloric acid yields furfural at 100°. Washed starch, in which the absence of pentosans was proved by means of phloroglucinol and hydrochloric acid, evolved notable quantities (0·44 to 1·16 per cent.) of furfural on boiling with hydrochloric acid (specific gravity, 1·06). Under similar conditions, pentosans themselves exhibit the same property. These experiments indicate that Tollens' process for estimating pentosans in hay is incorrect unless all sugar-forming substances (starch, dextrocellulose, etc.) are removed before treatment with acid.

F. H. L.

Estimation of Pentoses in Presence of Saccharose. Andrlik. (*Böhm. Zeits. Zuckerind.*, 1899, xxiii., 314; through *Chem. Zeit., Rep.*, 1899, 84.)—When pentoses are estimated by the phloroglucinol method, as the amount of sugar rises, increasing errors are produced, until 0·6 to 0·9 per cent. too much pentose is returned. The author has prepared a curve, by means of which the figures obtained on analysing mixtures of 9 grammes of saccharose with up to 3 per cent. of arabinose can be corrected. Per 100 parts of sugar, the diffusion syrup contains 0·20, the concentrated syrup 0·13, the first crystals 0·03 of pentoses; the remainder passing into the sludge and the molasses. F. H. L.

Influence of Temperature on the Optical Determination of Sugar. — Herzfeld. (*Zeits. Zuckerind.*, 1899, xlix., 1; through *Chem. Zeit. Rep.*, 1899, 65.) — Polarimetric observations ought to be made in a room where the temperature has remained constant for three hours; and in every laboratory a definite working temperature should be adopted, at which the value of the quartz plates should be checked on the normal weight of pure sugar. Correct results can thus be afterwards obtained with a half-shadow apparatus, even if the temperature of the test differs from the standard; but to be strictly accurate a correction of 0·00036 for every degree of variation from the 100-point and from the normal temperature should be made when working on crude sugars if the divergence in temperature is serious.

A simple setting of the zero point in good polarimeters insures the accuracy of the scale at the temperature of correction (17·5° or 20°); at other temperatures the value of the graduations changes. A normal sugar solution prepared at 20° and examined between 22° and 35° shows losses in rotatory power up to 0·6 per cent., and similar differences occur even if the tubes are not at the proper temperature.

F. H. L.

Methods for Distinguishing between Raw Linseed and Boiled Oil. F. Evers. (*Chem. Zeit.*, 1899, xxiii., 334.)—Linseed oil which has been boiled in presence of a drier is usually browner in colour, different in odour, and quicker in drying than the original material; but these tests are not certain, and are not available for the identification of mixtures of raw and boiled linseed oil. The author finds a process employed by the German Customs authorities quite satisfactory: 5 c.c. of a solution of 100 grammes of crystallized lead acetate in 150 c.c. of water are mixed with 1 c.c. of 20 per cent. of ammonia and 32 grammes of anhydrous glycerin; 12 c.c. of the oil are agitated with 6 c.c. of this reagent, and heated on the water-bath for three minutes. Raw linseed oil yields two distinct layers of liquid, the lower of which is perfectly clear; boiled oil containing a drier gives an emulsion. The metallic driers can also be extracted from a boiled oil by agitation with boiling dilute nitric acid, and the aqueous liquid can be examined as usual.

Linseed-oil "varnish"—*i.e.*, an oil boiled at a high temperature, or treated at a lower temperature with oxygen, without any addition of driers [*e.g.*, "lithographic varnish"]—does not respond to these tests, but naturally behaves like raw linseed. Morpurgo's process, however, is trustworthy. It depends on the fact that the soaps of raw linseed oil can be completely salted out of their aqueous solution, whereas those from a lithographic varnish remain partly in the liquid. The soap solution is prepared by the ordinary method of alcoholic saponification, the spirit is driven off, and the clear dilute liquid is treated with common salt as long as a precipitate falls. The mass is filtered, and acetic acid added in large excess: turbidity indicates an oil boiled without driers.

F. H. L.

[It may be remarked that linseed oil is seldom boiled without driers, except to such an extent as to raise considerably the specific gravity, redden the colour, cause a green fluorescence, and (mostly) to increase the viscosity to the eye. "Oxygenated" oils are not generally fluorescent, nor appreciably different in shade. In all cases, however, the iodine absorption is greatly diminished, falling some thirty to fifty points during the earliest stages of the boiling or blowing process. Oxygenated oils have a more disagreeable odour, especially on warming, than such as are prepared by fire heat; their specific gravity is higher; and they tend to dry rather more rapidly. However manufactured, the less viscid descriptions of lithographic varnish do not dry at all quicker than raw oil; while as the boiling progresses, the drying power (actually or apparently) falls off. *Cf. J. Soc. Chem. Ind.*, 1894, p. 203.—Abs.]

Examination of Oils and Varnish with the Refractometer. M. Weger. (*Zeits. angew. Chem.*, 1899, 297 and 330.)—In 1895 Hefelmann and Mann stated

that the purity of an oil or oil-varnish could be ascertained by examining the products in Zeiss's refractometer, and that the extreme limits were 80·6° to 84·2° for genuine varnishes uncontaminated with rosin, rosin oil, or mineral oil. Further experiments have shown that, although this is true in the majority of cases, there are numerous exceptions and irregularities, so that the optical method is not trustworthy unless it is checked by a chemical investigation—*e.g.*, determination of the saponification value and the amount of unsaponifiable matter.

As regards pure linseed oil, the refractometer number at 25° in sodium light ranges between 80·2° and 82·2°, one English specimen, which possessed an unusually high iodine number and a great capacity for absorbing oxygen, giving 85·1°. After "boiling," blowing with air, or the natural absorption of oxygen on keeping, linseed oil has a higher refractometer number; but the several products cannot be distinguished one from another by this means. The quantity of oxygen that a pure linseed oil or varnish is capable of absorbing rises with an increasing iodine number, but the refractive power does not invariably rise in the same manner—at least, such parallelism is only characteristic of new, raw oils. Among the boiled and oxidized oils and linseed varnishes the oxygen absorption and iodine values stand in inverse proportion to the refractometer number. The general effect on the refraction of linseed oil of treating it in various ways is shown in the annexed table; and similar results obtain with the non-drying oils, raw rape showing 69·2°, blown or thickened rape, 74·7° and 78·2°.

Sample No.	Material.	Refractometer No.
1	Raw Indian linseed oil	80·2
2	No. 1 + 1 per cent. of mangano-lead resinate, prepared cold ...	81·6
3	,, + 2 ,, ,, ,, ...	82·8
4	,, + 3 ,, ,, ,, ...	83·6
5	,, + 5 ,, ,, ,, ...	85·8
6	No. 6 heated to 150° C. for 1 hour	86·7
7	No. 1 + 1 per cent. of manganese linoleate dissolved at 50° ...	81·1
8	No. 7 heated to 150° C. for 2 hours	82·2
9	No. 1 + 0·5 per cent. of litharge and 0·3 per cent. manganese hydroxide heated to 230° C. for 2 hours	85·5
10	Commercial "Elektrofirniss," free from driers	89·8
11	English "ozonized" varnish containing 2 per cent. mangano-lead resinate	87·3

The refractometer numbers given by different mineral oils vary from 67·1° to over 105°; rosin and rosin oil also exhibit figures higher than 105°; fresh American oil of turpentine shows 65·5°, and, after exposure to air and partial evaporation, 77·0°. Therefore the refractometer cannot ensure the absence of these substances; and if they be not present, a high optical number does not point to adulteration, but may even be evidence of good qualities. The most that can safely be said is that an oil giving figures lying between Hefelmann's limits must be genuine. Weger's article includes sundry tables recording the refraction, oxygen-absorbing power, speed of drying, etc., of oils, boiled oils, drying oils, and linseed varnishes, made by different processes, and examined fresh or after being stored for various intervals of time.

F. H. L.

Analysis of Dégras, or Sod-Oil. E. Hopkins, D. L. Coburn, and E. Spiller. (*Journ. Amer. Chem. Soc.*, 1899, xxi., 291.)—Dégras proper is defined as the oil obtained from skins in the manufacture of chamois leather, as distinguished from sod-oil, which is the oil obtained from ordinary leather. In its preparation cheap fish-oils are mostly used—*e.g.*, menhaden and cod oils; but even olive-oil and certain greases are occasionally employed, which give the "constants" an exceedingly wide range. The chief features of dégras are the presence of the so-called "dégras former," hide fragments, and a high proportion of ash, and it is claimed that the value of the material rises with the amount of "former" and diminishes with that of mineral acid; but the price of the product does not appear to be governed by these factors. Dégras former, a comparatively uninvestigated substance, is described as being of a resinous nature (not a resin), melting at 65° to 67° C., and showing certain degrees of solubility in various solvents.

To estimate the moisture, the authors prefer to absorb the oil in a roll of ashless paper, enclosed in a folded filter (like a paper capsule), placed between two wads of cotton in a short test-tube, with a hole at the base. This is dried to constant weight at 100° C. while a current of air is aspirated through it. The dried sample is extracted in a small Soxhlet with petroleum ether (boiling-point 40° C.) to give the soluble matter, then with alcohol to recover the soap, while the residue is ignited to yield the ash and (by difference) the hide fragments. As both soap and oil contain inorganic matter, the total ash should be determined on another sample. The dégras former is estimated by a modification of the Jean process: The oil is saponified (determining unsaponifiable matter at the same time), the solvents evaporated, the soaps dissolved in hot water and salted out, the liquid filtered, cooled, and hydrochloric acid added; the former is collected on a filter (simple decantation leads to loss), washed with water till free from salt, etc., and with petroleum spirit till free from oil, then dissolved in absolute alcohol, evaporated, and weighed. It should melt at 65° to 67° C. As it exists in sod-oil, dégras former can be extracted by petroleum spirit, but in the pure state it is insoluble in this reagent. When titrating dégras soaps with acid in presence of phenolphthalein to ascertain the saponification value, etc., the interference of the dark liquid may be avoided by shaking the flask and watching the appearance of the foam.

The paper also includes copious tables of the results obtained on analysing some twelve different specimens of dégras; but in view of their very irregular and divergent character, the figures are scarcely capable of useful reproduction or condensation. The proportion of dégras former ranges between 0·65 and 21·41 per cent.; of hide fragments between 0·15 and 3 per cent.; of mineral acid in terms of milligrammes of potassium hydroxide per gramme of oil, between 0·96 and 90·5 per cent.; and of total ash between 0·05 and 1·05 per cent.—all calculated on the original wet material.

F. H. L.

Estimation of Tannins, and the Influence of Gallic Acid on the Process. A. Heinemann. (*Zeits. angew. Chem.*, 1899, 245.)—This article consists of a comparative study of the von Schröder-Löwenthal volumetric and the Simand-Weiss gravimetric methods of estimating tannins, as well as an investigation into the effect

produced on the results by the presence of varying proportions of gallic acid. The paper is scarcely capable of systematic abstraction; but the following points are brought out in the course of the work. No processes except those mentioned above are of the least use in valuing materials containing tannin; Vignon's idea of employing silk instead of hide-powder may perhaps be equally practicable; no process whatever can lay claim to real accuracy. The two chief sources of error in the hide-powder methods are (*a*) the undesirable precipitation of various substances (colouring matters, gallic acid) which were previously held in solution by the tannins, but which are no longer soluble after the tannin-hide reaction has taken place, and (*b*) the absorbing power of the hide-powder for non-tannins. As the solutions used in the volumetric process are less concentrated than those in the gravimetric, *a* is not so serious in the former case; on the contrary, for the same reason, and on account of the larger excess of hide-powder (30 : 1, as against 3 : 1), *b* is the chief defect of the volumetric method. This may partly be avoided by conducting the tannin absorption in stronger liquids (1 : 100), and diluting to 1 : 500 before titration. When the two processes yield different results, the gravimetric one is always lower, and probably more correct. The difference can only be explained by the absorption of bodies other than tannin which reduce permanganate powerfully. The difference, also, is roughly proportional to the amount of gallic acid present.

Mixtures of tannins with gallic acid cannot be valued by the volumetric process, for 1 gramme of the dry acid reacts with the same quantity of permanganate as 1·505 gramme of dry tannin. Provided the solution does not contain more than 5 or 10 per cent. of gallic acid, the gravimetric process is fairly trustworthy, each 1 per cent. of gallic acid up to 15 per cent. increasing the yield of tannin by 0·19 per cent., and from 15 to 30 per cent. by 0·17 per cent. A large excess of hide-powder in a dilute liquid absorbs almost all the gallic acid; 3 grammes per 1 gramme of tannin in 100 c.c. is sufficient.

The Weiss-Eitner-Simand process (*Zeits. Anal. Chem.*, xxviii. 110) is not satisfactory; and it must be remembered that the effect of adding 1 gramme of hide-powder three times in succession, and filtering between each, as done by Weiss, is widely different from that of introducing 3 grammes at once. The former plan leads to increased absorption of gallic acid. Gravimetrically, Vignon's method requires enormous weights of silk (11 : 1); apart from this, the modification is fairly good. Volumetrically it exhibits all the defects of the von Schröder-Löwenthal process, except that it absorbs far less gallic acid.

On the whole, the following seems to be the best method of conducting the operation: The hide-powder is agitated with twenty times its weight of water, allowed to settle, and filtered through linen; this is repeated six or eight times, and finally it is extracted with alcohol, filtered, and dried at 100° C. The tannin solution should contain from 1 to 1·5 gramme per 100 c.c.; it is treated with 3 grammes of hide-powder, and allowed to react for fourteen or sixteen hours, with occasional agitation. The mass is filtered through paper, the filtrate tested for tannin with a 1 per cent. solution of gelatin containing (as a preservative) a trace of phenol, and, if thought necessary, with a solution of pure tannin for dissolved hide-powder. If neither is present, an aliquot portion is evaporated to obtain the non-tannins and the tannins by difference.

The author complains that hitherto analysts have always approached this problem from the leather manufacturer's point of view, whereas the value of a particular "tannin" is by no means the same to the tanner, the dyer, and the writing-ink maker. Gallic acid is simply an impurity in the two former businesses, but it is very serviceable in the preparation of an iron ink. The article concludes with sundry speculations as to the theoretical constitution of the tannins.

F. H. L.

Separation and Identification of Coumarin and Vanillin. W. H. Hess and A. B. Prescott. (*Journ. Amer. Chem. Soc.*, 1899, xxi., 256.)—Twenty-five to 100 grammes of the commercial extract are evaporated at 80° C., with periodical additions of water to keep the volume of liquid constant till the alcohol has volatilized. Normal lead acetate is dropped in with agitation as long as a precipitate falls; the mass is filtered under pressure through asbestos, and the precipitate washed with a few c.c. of hot water. The cool filtrate is extracted three or four times with ether, or preferably chloroform, till all soluble matter is recovered; and the solution is treated in a separating funnel several times with 2 c.c. of 1 : 2 ammonia as long as the aqueous liquid becomes yellow. The ether is washed with 2 c.c. of water (the washings being mixed with the ammonia), then allowed to evaporate spontaneously or over sulphuric acid *in vacuo*, using a sealed capillary tube with its open end beneath the solution to prevent bumping. When free from moisture, or after drying at a temperature not exceeding 45° C., the ethereal residue is dissolved in a little cold petroleum spirit (boiling point 30° to 40° C.), the solvent is driven off *in vacuo* over paraffin, the coumarin dried at or below 45° and weighed. Its melting-point (67°) should be determined, and, coupled with the odour, is sufficient for identification. The ammoniacal liquid containing the vanillin is rendered faintly acid with 10 per cent. hydrochloric acid, then shaken out with ether, the solvent removed, and the residue dried *in vacuo* over sulphuric acid, or at a temperature not exceeding 55° C. The mass is extracted with boiling petroleum spirit, the solution decanted into a tared dish, evaporated at 55° and weighed. The yield should be pure vanillin melting at 80° to 81° C. After weighing, its colour reactions may be tested; but it should be noted that some vanillin extracts contain phenol-like bodies that give colours with iron salts, therefore such tests are only trustworthy after purification. Experiments on artificially prepared mixtures gave yields of 96 to 98 per cent. of the coumarin taken. The process is quicker and easier to work than the bisulphite method, and the separation of the vanillin is more complete.

F. H. L.

The Hæmatin of the Blood of Different Animals. P. Cazeneuve and P. Breteau. (*Journ. Pharm. Chim.*, 1899, ix., 321-23.)—The authors find that hæmatin prepared in the following rapid manner is a chemically pure substance, and varies in composition with the species of animal from whose blood it was derived.

The defibrinated blood is heated to the boiling point with its own weight of sodium sulphate. The precipitate is collected, washed with boiling water, slightly drained, and extracted at 50° C. in a mortar with successive small portions of 93 per

cent. alcohol, containing 10 grammes of oxalic acid per litre. About 2 litres of solvent are required to extract the precipitate from 1 litre of blood. Strong ammonium hydroxide solution is added drop by drop to the filtered alcoholic extracts, care being taken that the liquid still remains acid. The gelatinous precipitate of hæmatin is collected on a filter, washed with cold alcohol, redissolved in dilute ammonium hydroxide (5 per cent.), and reprecipitated with acetic acid. Finally it is washed successively with cold water, alcohol (93 per cent.), and ether, and dried first at the ordinary temperature, and then for twenty-four hours at 135° C. The yield amounts to about 1 gramme of hæmatin from a litre of blood.

According to the authors' analyses, the hæmatin thus obtained from the blood of the ox, horse, and sheep has the following elementary composition:

	Ox.	Horse.	Sheep.
Carbon	64·68	64·37	64·24
Hydrogen	5·33	5·38	5·32
Nitrogen	9·02	10·11	9·41
Iron	8·81	9·38	10·65
Oxygen...	12·16	10·76	10·38
	100·00	100·00	100·00

An analysis of the hæmatin of ox-blood by Hoppe-Seyler is given for the purpose of comparison: Carbon, 64·30; hydrogen, 5·50; nitrogen, 9·20; iron, 8·83.

C. A. M.

Note on the Estimation of Hydrocyanic Acid by Liebig's Method. **Lextreit.** (*Journ. Pharm. Chim.*, 1899, ix., 323-25.)—Liebig's method of determining hydrocyanic acid consists in first neutralizing the acid with potassium hydroxide, and then titrating the liquid with a standard solution of silver nitrate. A very soluble double cyanide of silver and potassium is produced, and the end of the titration is shown by the formation of the insoluble silver cyanide. The author points out that there are two objections to this method. If any free hydrocyanic acid is left in the liquid it does not react with the silver nitrate. And on the other hand, if too large an excess of potassium hydroxide be added the formation of silver cyanide is interfered with. Moreover, alkaline cyanides themselves have a distinctly alkaline reaction towards ordinary indicators, such as turmeric and phenol-phthalein, and it is therefore difficult to determine the exact point of neutralization.

This difficulty is obviated by using as indicator the blue C4B, which is not acted upon by alkaline cyanides or hydrocyanic acid, but is changed to red by alkaline hydroxides.

Having determined the amount of alkali required to neutralize the acid in the liquid, this quantity is added to a similar portion, and the titration with silver nitrate carried out in the usual manner.

Medicinal hydrocyanic acid is frequently prepared by decomposing potassium cyanide with tartaric acid. Liebig's method is unsuitable for the valuation of such preparations, since silver tartrate is formed and disappears too slowly, especially towards the end of the reaction, so that the final reading is not sharp. C. A. M.

Estimation of Uric Acid. E. Mallet. (*Rep. Pharm.*, 1899, lv., 100; through *Chem. Zeit. Rep.*, 1899, 93.)—This is essentially a combination of the Denigès and the Hopkins processes. One hundred c.c. of urine are treated with 10 c.c. of a 16 per cent. solution of anhydrous sodium carbonate to remove the phosphates. The filtrate is mixed with 5 c.c. of a 4 per cent. solution of copper sulphate and 20 c.c. of a reagent prepared as follows: sodium thiosulphate (cryst.) 100 grammes, Rochelle salt 100 grammes, water 1 litre. The precipitate is well washed, suspended in 500 c.c. of water, decomposed with 5 c.c, of sulphuric acid, and the uric acid titrated with permanganate. The results agree well with those obtained by the methods referred to above. F. H. L.

Reactions between Iron Salts and Pyrogallol. A. Hirsh. (*Pharm. Zeit.*, 1899, xliv., 205; through *Chem. Zeit. Rep.*, 1899, 99.)—When a few c.c. of a 1 per cent. solution of pyrogallol are mixed with 2 drops of dilute ferric chloride, an amethyst blue colour is produced, which very quickly changes to reddish-brown. One c.c. of 0·1 per cent. sodium hydroxide solution alters the colour to dark blue, and a trace of mineral acid restores the brown. The brown tint is, therefore, due essentially to the inorganic acid; for on adding ferric acetate to the pyrogallol, a blue colour is formed, which is also changed to red-brown by a mineral acid. With ferrous salts the white turbidity mentioned by Beilstein does not appear if the solutions are very weak, and an intense blue coloration is given on the addition of a minute amount of caustic soda. F. H. L.

INORGANIC ANALYSIS.

Estimation of Sulphuric Acid in Presence of Iron. Determination of Pyrrhotite in Pyrites. O. Herting. *Zeits. angew. Chem.*, 1899, 274.)—Provided the instructions are carefully followed, Herting has always found Lunge's process quite accurate; and comparative experiments with Küster and Thiel's methods (ANALYST, this vol., p. 137) show that the latter are less rapid and convenient.

The author also calls attention to the frequent presence of pyrrhotite in American pyrites, which is not completely burnt in the usual furnaces, and refers to Cone's process (ANALYST, 1893, xxi., 223) for its estimation. F. H. L.

Estimation of Sulphuric Acid in Presence of Iron. G. Lunge. (*Zeits. anorg. Chem.*, 1898, xix., 454.)—This is a protest against the inference contained in Küster and Thiel's article (ANALYST, this vol., p. 137) that Lunge's process is tedious and inaccurate. It can be carried out completely in two or three hours; whereas the very removal of the iron in the methods suggested by the former authors occupies four hours. As regards accuracy, Jannasch himself has admitted that the Lunge process is trustworthy (*J. pr. Chem.* [2], xl., 236). F. H. L.

Determination of Tungsten in Steel. G. Auchy. (*Journ. Amer. Chem. Soc.*, 1899, xxi., 239.)—In the estimation of tungsten in steel the amount of acid in

the liquid from which the tungstic acid and silica are filtered should be as small as possible; and the preceding evaporation to dryness to render them insoluble should take place in hydrochloric (not nitric) acid solution. Evaporation of a nitric acid solution does not lead to a complete separation of the tungsten; but in presence of hydrochloric acid, provided an excess be not employed, only one evaporation to total dryness is necessary. The best method of procedure is to dissolve 4 grammes* of the steel in dilute nitric acid, evaporating until a scum appears on the surface, adding 30 or 40 c.c. of strong HCl, evaporating to dryness and heating on the hot plate, dissolving in 40 c.c. of strong HCl, evaporating again until a scum forms, taking up in 5 c.c. of HCl and 15 c.c. of water, heating, diluting with 25 or 30 c.c. of hot water and filtering.

At least in the case of steels low in phosphorus, the tungstic acid need not be removed before estimating the former; but the yellow molybdate precipitate must not be allowed to stand more than 2 or 2½ hours. When neutralizing with ammonia, excess must be avoided, or tungstic acid will be thrown down and remain undissolved in the nitric acid subsequently introduced. The usual method of getting ferro-tungsten into solution depends on a preliminary roasting; the author has obtained unsatisfactory results when operating in this manner. He prefers to attack the alloy by heating it with strong nitric acid, adding strong hydrochloric acid by degrees, and then following the above scheme. A metallic residue is left consisting apparently of almost pure tungsten; but this is readily oxidized by ignition after it has been removed by filtration. F. H. L.

Some Reactions of Chloric, Bromic, and Iodic Acids. D. Vitali. (*Giorn. Farm. Trieste*, 1899, iv., 39; through *Chem. Zeit. Rep.*, 1899, 99.)—When a solution of manganous sulphate acidified with sulphuric acid is treated with a bromate, a fine violet colour is produced, which is due to a mixture of basic manganese sulphate and free bromine. The salts of chloric and iodic acids yield no colours. This reaction may be used as a means of determining manganese; for if the violet liquid is boiled for some time, the metal is finally precipitated as dioxide.

Salts of iodic acid are decomposed in the cold by hydroxylamine sulphate, iodine being liberated; bromates only behave similarly on warming; chlorates remain unreduced. An analogous reaction occurs with compounds of phenylhydrazine, but the iodine, instead of being set free, combines with the phenyl group, producing a red precipitate which presumably consists of triiodophenol.

Hypophosphorous acid decomposes iodates in the cold, liberating iodine at first and yielding finally a mixture of a phosphate and hydriodic acid; bromates are attacked on warming; chlorates not at all. F. H. L.

Titration of Persulphates. M. le Blanc and M. Eckardt. (*Zeits. Elektrochem.*, 1899, v., 355; through *Chem. Zeit. Rep.*, 1899, 62.)—Processes for determining the active oxygen in solutions of persulphates which depend on addition of

* If 3·9655 grammes be weighed out, the yield of WO_3 multiplied by 20 gives directly the percentage of tungsten in the steel.

ferrous ammonium sulphate, followed by titration with permanganate, are apt to give too low results, unless the temperature during the operation be raised. The employment of a large excess of iron salt serves the same purpose, but warming is simpler and more accurate. F. H. L.

A New Indicator for Acidimetry. E. Riegler. (*Bulet. Soc. de Sciinte din Bucuresci*, 1899, vii., 453.)—This body consists of the diazo colour which is prepared by treating an alkaline solution of guaiacol with diazoparanitraniline. It is a brown substance, insoluble in water, and soluble in alcohol. 0·20 gramme dissolved in 100 c.c. of spirit forms an indicator exceeding phenolphthalein in sensitiveness, bright red in alkaline liquids, greenish-yellow with acids. F. H. L.

REVIEWS.

CHEMICAL AND BIOLOGICAL ANALYSIS OF WATER. T. H. PEARMAIN and C. G. MOOR, M.A. Cantab. (Baillière, Tindall and Cox. Price 5s.)

In the 168 pages the authors have given a résumé of the subject which, if not sufficiently detailed for the practitioner, will undoubtedly be of service to the lay reader. The work is mainly a reprint from articles in the *Sanitary Record*, and presents a selection of processes which the authors have found convenient. Forty-seven pages are devoted to bacteriology and a chapter on aerated table-waters completes the volume. S. R.

THE MICROSCOPY OF DRINKING WATER. GEO. CHANDLER WHIPPLE, of the Brooklyn Department of Water Supply. (Wiley and Sons, New York; and Chapman and Hall, London. Price 3.50 dollars.)

So much exclusive attention is at present allotted to the Schizomycetes that the larger organisms in most text-books are passed over in a few paragraphs with rare and often incorrect illustrations, with the result that information has to be laboriously gathered from large and expensive monographs like those of M. C. Cooke and others. This book, then, with its delicately-executed plates, succinct descriptions, completed by a brief but sufficient glossary, and useful bibliography and tables, will be welcomed by the "water analyst and waterworks engineer," for whom it is intended. The latter is often painfully cognizant of the objectionable taste, odours, or appearances that many of the macro-organisms communicate to a supply. In Chapter XII. instances are given of the obstructions and difficulties that at recent dates these growths have occasioned in pipes and aqueducts, and "systematic and thorough flushing" is enjoined. The sections on distribution of minute life are exceedingly interesting, and seem exact; on p. 123 quantitative experiments are detailed, comparing the number of these organisms with definite amounts of various odorous chemicals. It is found that "in some cases the odoriferous substances from the organisms pass through the filter, and the disintegration of the organisms gives the filtered water an increased odour over the unfiltered." Though the work is modestly said to be "elementary

in character," and to have been prepared "during the leisure moments of a busy year," it is a valuable contribution to a somewhat neglected section of "Limnology" —the study of lakes and ponds. If any fault can be found, it is that while the Sedgwick-Rafter sand-plug and the "plankton net" of Hensen are fully described, the Dibdin micro-filter, which would appear for many reasons to be preferable, is not mentioned, and the Italian and German researches on the pathogenic action of some Protozoa should also have had some notice. The lesson of the book is summarized in the opening sentence of Chapter X.: "To obtain a permanently safe and satisfactory surface water supply without filtration, the rainfall must be collected quickly from a clean watershed and stored in a clean reservoir"—ideal conditions which are not always attainable in populous countries. S. R.

COMMERCIAL ORGANIC ANALYSIS. By ALFRED H. ALLEN. Third edition. Vol. II., Part I.: Oils, Fats, Waxes, Glycerol, Nitroglycerin, and Nitroglycerin Explosives. Revised and Edited by DR. HENRY LEFFMANN, Philadelphia. (London: J. and A. Churchill, 1899.) Price 14s.

Mr. Allen's great work on "Commercial Organic Analysis" is a text-book which has been found indispensable in every practical laboratory, and which is so well known that it needs no extended notice whenever a new edition of one or other of the volumes appears. We cannot refrain from congratulating the author that new editions are called for, for they give him the opportunity of bringing his work up to date, the more so as the literature on the technical subjects which are treated of by Mr. Allen is growing by leaps and bounds.

The labour of keeping a work of the magnitude of Mr. Allen's up to date is so enormous that one need not experience any surprise that the author has found it necessary to call in the aid of a collaborateur on the present occasion, although it is at the same time to be regretted that Mr. Allen has not been able personally to conduct the work comprised in the present volume, referring as it does to a branch of knowledge in which he is an acknowledged authority, and to which he has contributed so much original information. The aid of an American collaborateur has been felt necessary on account of the threatened appearance of an unauthorized and piratical American reprint, and the present volume therefore appears simultaneously in England and America.

The new edition contains many additions of new matter which has appeared during the last few years—bromine thermal methods, methods for the determination of glycerol, acetyl numbers, various tests for oxidation of oils, composition of and methods for the examination of dynamites and smokeless powders, dégras, cloth oils, and so forth.

Some of these additions are indicated by a different type from that used in the text of the book, while others appear as an appendix to the volume.

There is no class of organic substances of such immense technical importance in the investigation of which so much remains to be done as in that of oils and fats. Some advance in our knowledge has been made during the last ten or fifteen years, but the progress has been far from being in proportion to the importance of the subject.

The research chemist, who naturally prefers to work with compounds which admit of ready isolation and study, has deliberately kept his hands from unpromising and uncrystallizable unsaturated fatty matters. Almost everything which has of late years been accomplished in the elucidation of this theme has had to be done by practical chemists, upon whom the need for differentiating the multitude of oils occurring in commerce has daily pressed. Happily the empirical knowledge which was formerly deemed sufficient is slowly making place for more scientific views.

If we have any fault to find with the volume before us, it is perhaps that sufficient prominence has not been given to the *real* chemistry of oils, little as we know of it. If the problem of their constitution—a most complex and difficult one in any case—is to be solved as satisfactorily as that of sugars and other carbohydrates has been in recent years, it is obvious that new paths of investigation will have to be adopted, and the ripe experience of Mr. Allen in indicating such new paths would have been of great value to investigators.

As yet even the constitution of a body apparently so simple as oleic acid can hardly be regarded as definitely settled; we know nothing of the constitution of the less saturated acids, like linolenic acid; we are still in ignorance as to the constitutional difference between any two oils possessing approximately similar iodine absorptions but of widely different properties; and since we do not know whether these differences are due to constitutional differences in the fatty acids, or whether we have to do with different mixed glycerides, it is obvious that a vast field for research is still left practically untouched.

Since the appearance of the first edition of Mr. Allen's volume on fats and oils, the literature on the subject has increased rapidly. A vast amount of material has been collected which deals mainly with constants—admirable aids to the practical man, though only of moderate theoretical value. In comparing the present volume with the most comprehensive work on "Fats" by another author, it must not be forgotten that Mr. Allen's volume is a portion—only a small portion—of a great and comprehensive work travelling over a vast field of which fatty bodies form but a small part. Just as little as one would expect to find in a general text-book on chemistry a full account of all the modern knowledge, for instance, of the sugars, so Mr. Allen's work should be considered mainly from the point of view on which the whole work was undertaken—the detection and determination of the impurities, adulterations, and products of decomposition of the materials referred to in the work.

As a practical guide, Mr. Allen's third edition is of the same high standard of excellence as were the previous ones, and anyone who has to analyse fatty substances will find in it the fullest and most trustworthy practical information. O. H.

THE ANALYST.

JULY, 1899.

PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS.

THE monthly meeting of the Society was held on Wednesday evening, June 7, in the rooms of the Chemical Society, Burlington House. In the unavoidable absence of the President the chair was taken by Mr. Alfred H. Allen (Vice-President).

The minutes of the previous meeting were read and confirmed.

Certificates of proposal for election to membership in favour of the following candidates were read for the second time: Thomas Waterworth Glass, B.Sc. (Lond.); Henry Dixon Hewitt; Daniel Lewis Thomas; and Samuel Allinson Woodhead, B.Sc.; and in favour of the following candidates for the first time: Frederick Davis, Analyst, 51, Imperial Buildings, Ludgate Circus, London; William Francis, Chief Analyst to the Condensed Milk Company of Ireland, Limited, Limerick; and Edward Russell, B.Sc. (Lond.), F.I.C., Demonstrator of State Medicine, King's College, London.

The CHAIRMAN (Mr. Allen) said that he had recently paid a short visit to the United States, and wished to express to the Society his sense of the cordial and friendly feeling entertained by American chemists towards the Society of Public Analysts as a society, and towards its members individually—a feeling which he was sure would be heartily reciprocated.

The following papers were then read: "On some Comparative Analyses of, and Digestive Experiments with, White and Wholemeal Breads," by Otto Rosenheim, Ph.D., and P. Schidrowitz, Ph.D.; "Note on Coffee Extracts," by C. G. Moor, M.A., and Martin Priest; "On the Determination of the Iodine Value," by J. Lewkowitsch, Ph.D.; and "On the Influence of Ammonium Salts on the Precipitation of Nickel by Ammonia," by Arthur Marshall.

ON A RECENT GINGER CASE.

By E. J. BEVAN, BERNARD DYER, AND OTTO HEHNER.

(Read at the Meeting, April 12, 1899.)

IN a recent case heard at Barnsley, Mr. Allen certified that a certain sample of ginger consisted of 50 per cent. of genuine ginger and 50 per cent. of ginger deprived of its pungent principles. The case was referred to the Government analysts, whose certificate upon the sample was as follows:

"Government Laboratory, Clement's Inn Passage, Strand, London, W.C.

"The sample of ground ginger marked No. 421, and referred to in your letter of

the 23rd ult., was received here on the following day, securely sealed. We hereby certify that we have analysed the ginger, and declare the results of our analysis to be as follows:

Soluble in 90 per cent. alcohol	4·56 per cent.
Soluble in ether (volatile oil)	0·43 ,,
Fixed oil and resin	4·43 ,,
Soluble in alcohol after ether	1·08 ,,
Soluble in water after alcohol and ether	3·70 ,,
Total ash	2·33 ,,
Ash soluble in water	1·03 ,,
Loss on drying sample (at 212° F.)	12·70 ,,

The microscopic and physical characters of the sample indicate that it belongs to one of the lower-grade descriptions of commercial ginger. The amounts soluble in alcohol and ether respectively are equal to that found in genuine ginger of this class, and afford no evidence that the sample has been exhausted with alcohol. The proportions of matter soluble in water, and of ash soluble in water, are lower than we have found in unwashed ginger; but as these amounts are easily reduced by the ordinary washing of the ginger, which usually occurs before grinding, they do not, in our opinion, afford evidence that the sample in question is a mixture of genuine ground ginger and ginger exhausted of its pungent principles. The microscopic and analytical results are consistent with the conclusion that the sample in question is a 'washed' Japan, or similar low-grade ginger. As witness our hands this eleventh day of February, 1899.

(Signed) "G. LEWIN, H. W. DAVIS."

It is quite possible that the supposition that this was a washed ginger may be correct; but if so, it had been washed in no "ordinary" fashion, but in such an exhaustive and drastic way that the matter soluble in water after alcohol and ether was reduced to 3·7 per cent. Of course, some of the matter soluble in alcohol after ether would be also soluble in water, but the total matter soluble in water was rather less than 5 per cent.

Even samples of ginger that have been washed more than is usual sometimes contain 9 or 10 per cent. of matter soluble in water, and no ginger that has been properly washed should contain less than 9 or 10 per cent. of matter soluble in cold water. It is perfectly clear that at least half the total matter soluble in water in this ginger had been removed.

The magistrates felt obliged to dismiss the case, for it was, of course, admitted, both by Mr. Allen and by ourselves (who gave corroborative evidence as to the exhaustion), that it was not possible to say whether the sample was a mixture, as literally described in the certificate, containing one-half of its weight of exhausted ginger, or whether it consisted entirely of half-exhausted ginger. If the certificate had been to the effect that half of the principles of the ginger had been exhausted, there would probably have been a conviction. Although the magistrates dismissed the summons, they stated, through their chairman, that the case was a perfectly

proper one to have been brought forward and proceeded with, and they refused to allow any costs to the defendant.

The main point of interest in the case to Public Analysts is the finding of the Government chemists, and the way in which it is expressed. It was clearly admitted in this certificate that washing to some extent removed the soluble matter, but there was no indication in the certificate that the washing (if the cause of the abnormality) had been in this case of an unusually drastic or exhaustive character, and no statement to the effect that the result of whatever had happened to this sample was precisely the same as if the sample had consisted of a mixture of exhausted and unexhausted ginger. Furthermore, the Government chemists appear to assume that exhausted ginger means ginger exhausted with alcohol, forgetful of the fact that fermentation ginger-beer is made by the water-exhaustion of ginger.

We have ourselves met with many samples of badly-washed ginger, but never with a case carried so far as this, assuming the exhaustion to have been produced as the Government chemists suggest. Ginger can be quite properly washed without any appreciable exhaustion; but it is washed by persons called in the trade "ginger-washers," who in their operations sometimes use hydrochloric acid and bleaching-powder, which have to be washed out; and if the washers steep the ginger for two or three days, it is obvious that they may turn out exhausted ginger.

From what we hear in the spice trade, we think there is some hope that the process of washing may be abandoned altogether, on account of the excessive extent to which it has sometimes been carried. Obviously it matters nothing to the ultimate consumer of the ginger how or by whom or for what purpose the exhaustion is produced.

Discussion.

The President (Mr. Fisher) said it was very unsatisfactory that an article like the sample referred to—which obviously must be regarded as exhausted ginger, by whatever means the exhaustion had been arrived at—should be passed by a certificate describing it as nothing worse than a low-grade ginger. That, he thought, was rather an unhappy expression, and it did not seem from the report that the authorities who issued it were particularly anxious to put down this tampering with ginger. Whether the exhaustion was wilful, or whether it arose through negligence or ignorance, could not be established; but it was regrettable that such a certificate should come from the principal analytical laboratory in the country.

Mr. Moor referred again to the question whether the British Pharmacopœia could be taken as laying down a standard in the case of ginger. The Pharmacopœia did not recognise washing at all, and he thought it was doubtful how far such trade customs ought to be regarded as being any excuse for abnormal samples.

Mr. Chattaway agreed with the authors that the probable result of the action which from time to time had been taken in this matter would be the use of unwashed ginger for grinding. He had gone into the matter pretty closely from a trade point of view, and had reason to think that there was very little ground ginger being made at present except from ginger that had only been washed at the port of export. On the other hand, almost all ginger that was sold whole was still washed at least once in this country, and probably also abroad before exportation.

THE ESTIMATION OF FAT IN MILK, USING PETROLEUM ETHER AS A SOLVENT.

By H. Droop Richmond and C. H. Rosier.

(*Read at the Meeting, April* 12, 1899.)

The almost universally used solvent for fat in milk analysis is ether, and the form usually employed in this country is methylated ether. This solvent has certain undesirable qualities, as it is composed largely of methyl-ethoxide, which, being very volatile, causes a considerable loss of solvent.

Petroleum ether is free from these drawbacks, and has the further advantage of being an excellent solvent for fats, and not dissolving many other substances. Another property in which petroleum ether differs from ether is its insolubility in water, a property which has been considered as an advantage, but which is the one property to which many failures to successfully employ this solvent is due.

In order to see plainly the conditions under which petroleum ether can be used to extract fat from milk, it is necessary to consider slightly the theoretical bearings of the subject.

Milk may be considered for this purpose as an emulsion of small fat globules in a somewhat viscous liquid; the relation between the surface energy of the fat and the liquid is such that a layer is condensed around the fat-globules, which prevents its being touched by anything except the liquid, unless special forces are brought into play. It would therefore be quite hopeless to expect to extract fat from milk by petroleum ether or any solvent which does not dissolve in the liquid portion.

By so modifying the relation between the surface energy of the fat and the liquid that the layer becomes a semi-permeable membrane, a solvent such as petroleum ether would extract fat from this liquid, provided the solvent could be brought into contact with each globule. A solvent like ether soluble in the liquid would be very much more efficient in this respect, as it would dissolve in the liquid, and distribute itself between liquid and fat in proportion to its solubility in each; as ether is very soluble in (liquid) fat, and fairly so in aqueous liquids, the fat globules would soon take up large amounts of ether, and become practically solutions of fat in ether; from their large size and low density they would rapidly rise to the surface, and coalesce to form a liquid layer, and the fat would without difficulty become evenly distributed throughout the ether on shaking.

To attain the same end with petroleum ether, the mixture must be so perfect that every fat globule is brought into contact with petroleum ether. This state of things can only be attained in practice when a sufficient length of time is allowed for the fat globules to rise to the surface, which would prolong the time necessary for an estimation of fat to many hours. Under these circumstances the use of petroleum ether would be a disadvantage.

The employment of amyl-alcohol to expedite the time taken for the fat to rise to the surface, devised by Leffmann and Beam, and elaborated by Gerber, appeared to be a condition likely to insure the extraction of fat by petroleum ether in a short time. We had no fear that amyl-alcohol would be extracted with the fat, as we were

convinced by previous experiment that it combined with the sulphuric acid used to make the milk suitable for extraction, and formed a compound very soluble in water.

The mode of procedure described below was devised entirely from theoretical considerations, though we hoped at first to be able to omit the washing with water. The fact that the process founded on theory worked well in practice incidentally affords evidence of the correctness of the views set forth above.

The following experiments illustrate the slowness with which petroleum ether extracts fat from milk boiled with hydrochloric acid, when nothing is present to hasten the separation of the fat. Three samples of milk were treated by the Werner-Schmid process, petroleum ether being substituted for ether.

	I.	II.	III.
Percentage of fat (Storch)	3·95	3·74	3·84
Fat deduced from the evaporation of an aliquot portion of petroleum ether	—	2·28	—
Fat in three extractions with petroleum ether ...	2·59	3·20	2·83
Fat in a second three extractions with petroleum ether	2·84	3·31	2·94

We next substituted sulphuric acid for the hydrochloric acid, and added amyl-alcohol—in fact, we used the Gerber method as far as mixing was concerned, but instead of placing in a centrifugal machine we cooled and added petroleum ether. This was allowed to separate, re-mixed, allowed to separate again, re-mixed a second time, and finally allowed to separate; an aliquot portion was evaporated. It was observed that the petroleum ether, after the first separation, was not homogeneous, a more highly-refracting layer being easily seen at the bottom. This was expected, and the re-mixing was adopted to guard against this. We found that the layer of petroleum ether containing fat always occupied a volume greater than the petroleum ether added, and the excess was practically equal to the volume of the fat. It was found also that petroleum ether extracted from a mixture of sulphuric acid, water, and amyl-alcohol, a small amount of substance having an ethereal smell.

The table below gives the results of our preliminary experiments:

	I.	II.	III.
Percentage of fat (Storch)... ...	3·34	3·88	3·88
Percentage of fat deduced from an aliquot portion, neglecting volume of fat...	3·38	3·91 3·88 3·87	3·91 3·88
Percentage of fat deduced from an aliquot portion, correcting for volume of fat	3·43	3·97 3·96 3·95	3·97 3·96
Percentage of fat deduced from an aliquot portion, correcting for volume of fat, and for blank ...	3·33	3·87 3·87 3·86	3·87 3·87
Percentage of fat deduced from complete extraction	3·52		
Ditto corrected for blank	3·32		
Percentage of fat deduced from complete extraction and washing the petroleum ether once with water...	—	3·90	3·86

In all experiments where the petroleum ether was not washed the fat darkened on drying, and an odour resembling that of amyl butyrate was developed; this we attribute to the extraction of a little amyl-hydrogen sulphate.

We found that when the petroleum ether was washed the fat was nearly white and odourless.

The following mode of procedure is now adopted: Nine c.c. of sulphuric acid (90-91 per cent. H_2SO_4) are measured into a tube holding about 50 c.c., and constricted just above the point where 20 c.c. reach; 10 grammes of milk are weighed into this tube, care being taken to prevent the milk and acid mixing; 0·9 c.c of amyl alcohol is added, the tube corked, and well shaken; after cooling to about 25° C., 20 c.c. of petroleum ether are added, and the tube well shaken. When separation is complete, the contents of the tube are again well mixed, and allowed to separate; a second re-mixture and separation is given, and the petroleum ether blown off into a tube containing 20 c.c. of water, with which it is shaken and allowed to separate. After separation from the water, the petroleum ether is blown off into a tared flask. Further portions of petroleum ether are added to the tube containing the acid liquid, blown off into the tube containing the water, and transferred to the flask.

We are in the habit of whirling the tubes in a Leffmann-Beam centrifugal machine to reduce the time necessary for separation.

The following additional results will show that this method compares well with others.

Petroleum Ether.	Other Methods.	Methods Used.
3·76	3·75	Storch
3·62	3·62	,,
3·04	3·00	Ritthausen
3·63	3·59	Storch
3·64	3·60	,,
3·67	3·66	,,
4·00	4·00	,,
64·72	64·70	Amyl alcohol extraction

We use this method chiefly because the employment of ether is avoided.

Discussion.

Dr Dyer thought that it was rather to the credit of petroleum ether that it did not give, in the Werner-Schmid process, the same good results which were obtained with ordinary ether. The Werner-Schmid method, although it gave excellent results, only did so in virtue of its errors being balanced in different directions. Scientifically speaking, in spite of its good results, it was a slipshod process. It was assumed that all the fat was obtained in the supernatant ether, but as a matter of fact a tangible proportion of fat remained in association with the ether that still remained in the acid fluid. However, the residue of the supernatant ether, which was called fat, contained, besides fat, nondescript substances, formed probably by the action of the acid on the milk sugar and curd. These impurities, by some happy natural provision, balanced the fat that was not dissolved in the supernatant ether, so that fairly accurate results were obtained. Petroleum ether apparently did not dissolve these substances, though after prolonged shaking higher results were obtained. The

Werner-Schmid process gave admirable results if it was carried out in a separator, the acid liquor being run off and washed with ether a second time, the total ether washings being then washed with a small quantity of water. In the ordinary way, if the ethereal residue were re-dissolved in ether after it was dry, there was a considerable quantity of material which did not dissolve. The errors, perhaps, were not very great, and balanced one another pretty closely in the case of ordinary milk, but in the case of separated milk the percentage error, having regard to the small proportion of fat, was often very large in the Werner-Schmid process as generally used.

Mr. CHAPMAN remarked that petroleum ether was somewhat variable in character, and inquired whether the particular kind of petroleum ether employed affected the results.

Mr. RICHMOND said that Mr. Rosier and he had only employed one kind of petroleum spirit, which was obtained by careful fractionation, and boiled below 80° C. As low a boiling-point as possible was chosen in order that the petroleum spirit might be readily driven off from the fat by drying in a water-bath. He might mention that the fat extracted by petroleum spirit was obtained perfectly white and free from odour if the spirit containing the fat were washed with water. If this were not done, a small quantity of acid was dissolved out, probably in the form of acid ethereal sulphate.

THE ASSAY OF BELLADONNA PLASTER.

By F. C. J. BIRD.

(Read at the Meeting, May 3, 1899.)

THE determination of the alkaloidal value of samples of the belladonna plaster of the Pharmacopœia is an operation which has presented some difficulty on account of the interference of the soap, resin, and other ingredients which enter into its composition. Belladonna plaster (B.P.) should contain 0·5 per cent. of the alkaloids of belladonna root (atropine and hyoscyamine), and there are present in addition lead plaster, resin, and hard soap, together with the evaporated residue of the liquid extract, consisting of fatty and resinous bodies, colouring and extractive matters. During the process of manufacture a certain amount of evaporation takes place, so that the alkaloidal content of the plaster should really be a trifle over 0·5 per cent.

Solvents such as petroleum spirit, chloroform, etc., were at first tried, but they failed to completely dissolve the plaster, and the attempt to wash out the alkaloids merely resulted in hopeless emulsification. Finally a mixture of glacial acetic acid and chloroform was found to be the most satisfactory solvent, only a small and negligiable quantity of colouring and extractive matter remaining undissolved. On addition of dilute sulphuric acid the lead oleate, stearate and soap are decomposed, lead sulphate being precipitated, and the liberated fatty acids and resin going into solution in the chloroform. The alkaloids as sulphates are taken up by the aqueous liquid, and if the whole be thrown on a Buchner's vacuum filter the lead sulphate can be removed as a solid cake, whilst the chloroform and aqueous layer pass through in a clear and easily separable condition.

The following is the process in detail:

Belladonna plaster (B.P.)	15 grammes.
Chloroform	35 c.c.
Glacial acetic acid	5 c.c.

Warm gently to dissolve, and add

Water	40 c.c.
Dilute sulphuric acid (1 in 12)	35 c.c.

Again warm and pour on a 4-inch Buchner's filter connected with a vacuum of 8 to 10 inches of mercury. Remove the nearly dry cake of lead sulphate, break it up, and warm with

Chloroform	10 c.c.
Dilute sulphuric acid	5 c.c.
Water	10 c.c.

Again filter, and transfer the mixed filtrates to a separator. Run off the chloroform (aiding its separation, if necessary, by warming), wash twice with

Dilute sulphuric acid	1 c.c.
Hot water	4 c.c.

and return the washings to the aqueous portion.

To the mixed aqueous liquids add

Chloroform	20 c.c.
Solution of ammonia	A decided excess.

Warm gently, and agitate. Continue the process according to the directions given in the Pharmacopœia, as follows, assisting the separation of the chloroformic layer, whenever necessary, by the application of a gentle heat: "Separate the chloroformic solution, and twice repeat the agitation with 10 c.c. of chloroform and the separation. Shake the mixed chloroformic solutions with 5 c.c. of diluted sulphuric acid (1 in 12), mixed with twice its volume of warm water; separate the chloroformic liquid, and repeat the agitation with acidulated water. Wash the mixed acid liquids with 3 c.c. of chloroform; then agitate with 10 c.c. of chloroform and an excess of solution of ammonia. Separate the chloroformic solution; twice repeat the agitation with chloroform and the separation; wash the mixed chloroformic solutions with 5 c.c. of water containing one drop of solution of ammonia; draw off the chloroformic layer into a counterpoised dish, evaporate on a water-bath, dry the residue below 100° C., and weigh. Dissolve the residue in 10 c.c. of a decinormal solution of hydrochloric acid, and add centinormal solution of soda, free from carbonate, until the liquid is neutral, using tincture of cochineal as indicator. Deduct the measure of the soda solution thus required from 100 c.c., and multiply the remainder by 0·00287; the product will be the weight in grammes of alkaloids present in the 15 grammes of plaster operated upon." The figures obtained by weighing and by titration should agree within about 5 milligrammes.

This method has been in use, with satisfactory results, during the past three months, and its accuracy has been verified by the analysis of samples of known composition.

DISCUSSION.

Dr. RIDEAL said that a considerable number of analyses of belladonna plaster had recently been made in the United States and Canada, where the subject was being much discussed. In America belladonna plasters were made with a rubber base instead of resin plaster, and the process of extracting the alkaloids from them was of necessity different from that required in the case of B.P. plaster. In the last edition of the British Pharmacopœia, under the heading "Emplastrum Belladonnæ," directions were given for preparing the plaster with a certain proportion of the standardized extract of belladonna, containing 0·75 per cent. of alkaloids, it being stated in addition that the plaster so made should contain 0·5 per cent. of alkaloids. Now, the alkaloids of belladonna were extremely unstable, and it was quite conceivable that the method of manufacture laid down in the Pharmacopœia might involve some loss of alkaloids. Since the publication of the new edition of the Pharmacopœia, the efforts of manufacturers have been directed towards the production of a plaster showing on analysis 0·5 per cent. of alkaloids, and he believed that considerable difficulty had been experienced in producing a 0·5 plaster from the 0·75 extract. Some manufacturers had, he believed, actually departed from the instructions, and used an extract stronger than that prescribed by the Pharmacopœia. He had had the opportunity of comparing Mr. Bird's process with some other processes, and had obtained some interesting results, examining for the purpose two specimens of plaster, respectively prepared by Mr. Bird's firm and by another firm according to the Pharmacopœia directions. In the first place he had estimated the alkaloid obtained by titration with Mayer's reagent, a process which was very generally used in the United States for the final valuation of belladonna plasters made with rubber. There was some difficulty in deciding upon the proper factor to be used with this reagent in the case of B.P. plaster, but he had eventually adopted that recommended by Dr. Muter, viz., 0·0049 for $\frac{N}{10}$ Mayer's reagent, which he believed was Dragendorff's original figure. This test gave 0·473 per cent. of alkaloids in the case of the plaster made by Mr. Bird's firm, while Mr. Bird's process (the separated alkaloids being weighed) gave 0·62 and 0·596 in duplicate determinations. In the latter cases, however, the alkaloids were probably not quite thoroughly dry. Titration with soda, according to the British Pharmacopœia, gave 0·512 or 0·518; and an American method, known as Smith's method, gave 0·454 per cent., a result which was obviously too low. The other sample gave 0·48 and 0·43 by Mr. Bird's process, 0·43 by the B.P. method, and 0·398 and 0·372 by Smith's process, the latter giving in this case also a result obviously too low. There was considerable advantage in a modification originally suggested by Smith. After the alkaloids had been obtained in a dry state, there was a little difficulty in getting them thoroughly dissolved in the acid before titrating; this difficulty was completely overcome by adding about 1 c.c. of chloroform to the solid alkaloids, so as to effect their solution before adding the acid. He thought hæmatoxylin, which was recommended by Smith as an indicator, was a little more satisfactory than the tincture of cochineal which was the official indicator in the B.P.

Mr. BIRD said it seemed only right to mention that about March last an

American journal published a process by Frank X. Moerk which was substantially the same in principle as that referred to in this paper, except that the plaster without previous solution was treated directly with aqueous sulphuric acid. This, however, was open to some objection, as it presented certain difficulties in working. He (Mr. Bird) had experimented with plasters of American origin, using glacial acetic acid and chloroform as a solvent, and had found that after about three minutes' immersion the whole of the rubber and belladonna was removed from the cloth. On treating the solution with sulphuric acid, there was very little difficulty in filtering it and getting a solution which responded very clearly to the tests for belladonna alkaloids; the method would therefore probably prove useful in the analysis of plasters with rubber bases. The process of manufacture might involve loss of alkaloids if proper precautions were not taken; but if the liquid extract were evaporated at as low a temperature as possible, and mixed with the resin plaster at a low temperature, there would be practically no loss. The first sample referred to by Dr. Rideal had rather an interesting history. It was made in strict accordance with the Pharmacopœia directions from a carefully assayed liquid extract of belladonna, with the view of ascertaining if loss of alkaloid was a necessary accompaniment of the B.P. process of manufacture, and when finally assayed showed 0·51 per cent. of alkaloids, a result almost in suspicious agreement with that obtained by Dr. Rideal in his analysis of the same sample by this process. The use of chloroform in taking up the alkaloids (as mentioned by Dr. Rideal) was sometimes absolutely necessary, for the alkaloids appeared to carry with them a film of grease, which greatly impeded the action of the acid. He had long since discarded cochineal, on account of the fact that in delicate determinations it was seriously affected by carbonic acid, and now only used methyl-orange as an indicator.

THE ESTIMATION OF PENTOSANS AND ITS APPLICATION TO THE ANALYSIS OF FOODS.

By Otto Hehner, F.I.C., and W. P. Skertchly, F.I.C.

(*Read at the Meeting, May* 3, 1899.)

It is well known, mainly owing to the researches of Tollens and his collaborators, De Chalmot, Stift, Stone, Cross, Bevan, and others, that many vegetable substances yield furfural on distillation with hydrochloric acid.

It is not our intention in this paper to discuss the origin of the furfural. This is attributed by Tollens and the majority of continental investigators to the decomposition of pentosans (anhydrides of penta-glucoses which yield xylose and arabinose on hydrolysis). Messrs. Cross and Bevan have, however, brought forward strong evidence that the production of furfural does not necessarily indicate the presence of the C_5 molecule, but is in many cases due to oxy-cellulose.

The carbohydrates of the C_6 type, or multiples of this, yield on hydrolysis with hydrochloric acid only insignificant amounts of furfural.

Without entering into the controversy as to the origin of the furfural, we have

followed the precedent of Tollens in the results given below, and have calculated the furfural into "pentosan."

Many determinations of furfural, and, by calculation from this, of the furfural-yielding pentosan, have been published during the last few years, mainly in feeding-stuffs intended for cattle, such as straw, hay, lupines, clover, and also in various kinds of wood in various stages of development. A good deal of evidence has also been accumulated which serves to show that the carbohydrates of the C_5 class either pass through the body without being assimilated, or at least are assimilated to a very much less degree than the hexoses and their multiples. From this it follows that it is a matter of considerable importance to distinguish between the C_5 and the C_6 types of carbohydrates in food substances, and that it is no longer permissible to take by difference "starch, sugar, and digestible fibre," as has been the practice for so many years.

But while a good deal of work has been done in food materials intended for animals, we can find but few data in literature to show that the determination of furfural-yielding bodies may also usefully be applied to articles of human consumption. Stone (*Journ. Amer. Chem. Soc.*, vol. xix., p. 195) gives a few determinations in whole wheats, bread, and various kinds of wheaten flour, which show that while whole wheat yields to 1 per cent. hydrochloric acid about 4·5 per cent. of pentosan, wheat flour (presumably free from bran) yields nothing. Stift (*Oest. Ung. Zeitsch. Zuckerind*, xxiv., 290) gives the percentage of pentosan in various articles as follows: Crushed barley, 7·96 per cent.; rice-meal, 5·73; broad beans, 3·43; turnips, 1·13; spinach, 1·02, etc.

The results of analyses which we bring forward in this paper tend to show that the determination of pentosan may in some cases be of considerable use to the analyst.

It is well known that the formation of furfural-yielding substances takes place mainly when lignification in the plant goes on. Hence in the case of seeds it might be expected that the husk would be rich and the kernel-substance poor in furfuroids.

The method of determination of furfural which we have followed is that of Councler; it depends upon the condensation of furfural with phloroglucinol, while the mode of splitting off the furfural is that of Tollens and Krüger (*Zeitsch. Rübenz. Ind.*, D.R., vol. xlvi., p. 480). The combined method, as described by König (*Untersuchung. landwirtschaftlich und gewerblich wichtiger Stoffe*, 1898, p. 225), is as follows:

From 3 to 4 grammes of the substance are placed in a Wurtz flask of about 250 c.c. capacity along with 100 c.c. dilute hydrochloric acid, of specific gravity 1·06, corresponding with 12 per cent. of real hydrochloric acid. The flask is connected with a condenser, and the contents are distilled on a sand-bath. When 30 c.c. of distillate have been collected, a further 30 c.c. are added to the distilling-flask through a tap-funnel, and this is repeated until 400 c.c. have distilled over. As the distillation proceeds, drops of the distillate are taken from the end of the condenser on filter-paper and tested for the presence of furfural by touching the drop on the paper with a dilute solution of aniline acetate containing sodium acetate. When a pink coloration no longer appears the distillation is stopped, and the distillate made up to 400 c.c. with the 1·06 hydrochloric acid; otherwise the distillation is carried on till

400 c.c. have been obtained. It will mostly be found necessary to distil over from 300 to 400 c.c. before the reaction reaches its end. A solution of pure phloroglucinol in dilute hydrochloric acid is now added to the distillate, and the mixture allowed to stand overnight. Next morning the black precipitate which forms is filtered off through a dried and weighed filter, and washed with 150 c.c. of cold water. The filter is laid upon a piece of blotting-paper, which absorbs superfluous water, is then opened out, dried in the water-oven for three and a half to four hours and weighed. The weight of the precipitate of furfural-phloroglucide divided by 1·82 gives the amount of furfural. Following the data given by Tollens, this furfural may be calculated into pentosan by subtracting 0·0104, and multiplying the remainder by 1·88. The percentage of the pentosan is calculated from the weight thus obtained.

This method has been tested by several observers, and found to afford an accurate determination of furfural if the phloroglucinol employed is pure. We have also made some experiments in this direction, with the following results: 1·1680 gramme of furfural was dissolved to 100 c.c. of water; 10 c.c. of this solution yielded 0·2112, and 0·2114 gramme of the phloroglucide, corresponding to 99·31 and 99·40 per cent. of the furfural taken.

In most of the analyses which follow the crude fibre has been estimated as well as the pentosan. As the determination of the fibre yields results largely dependent upon the strength of the acid and alkali employed, it should be stated that 5 per cent. hydrochloric acid and 5 per cent. sodium hydroxide solutions were used, the boiling with each solution lasting one hour.

Mustard.

	Pentosan.	Crude Fibre.
Flour of brown mustard ...	3·03 per cent.	2·94 per cent.
Flour of white mustard ...	3·33 ,,	3·83 ,,
Husk of white mustard seed ...	7·30 ,,	7·01 ,,
Mustard bran	9·52 ,,	10·95 ,,

The following are samples containing varying proportions of the brown and white seed:

Pentosan.	Crude Fibre.
2·92 per cent. ...	3·88 per cent.
2·77 ,, ...	1·92 ,,
0·76	2·77
0·89	3·26
2·63	2·77
3·59	2·76

Three samples mixed with wheat flour yielded results as under:

Pentosan.	Crude Fibre.
1·23 per cent. ...	3·18 per cent.
2·66 ,, ...	2·32 ,,
3·47 ,, ...	1·75 ,,

Coffee.

	Pentosan.	Crude Fibre.
Raw coffee	2·86 per cent. ...	—
Genuine roasted coffee ...	2·50 ,, ...	7·36 per cent.
Coffee containing 32 % chicory	2·71 ,, ...	—

Chicory.

These samples of chicory are all of one kind, but the samples were taken from the roaster after being roasted for different lengths of time. The results obtained from the above were as follows :

	Moisture.	Pentosan.	Crude Fibre.
Best high-dried Belgian root ...	5·51 per cent.	5·14 per cent.	5·47 per cent.
The same after 5 min. roasting ...	6·17 ,,	5·55 ,,	6·57 ,,
,, ,, 10 min. ,, ...	3·95 ,,	5·16 ,,	6·87 ,,
,, ,, 15 min. ,, ...	3·73 ,,	4·80 ,,	8·67 ,,
,, ,, 23 min. roasting, finished for grinding	3·28 ,,	5·56 ,,	11·50 ,,

Here it will be seen that "fibre" increases as the roasting proceeds, carbonized material being included with the fibre.

Pepper.

	Pentosan.	Crude Fibre.
Pepper husk	10·24 per cent. ...	14·68 per cent.
Black pepper	4·58 ,, ...	9·91 ,,
Genuine white pepper ...	1·68 ,, ...	5·54 ,,

Cocoa.

	Pentosan.	Crude Fibre.
Van Houten's cocoa ...	2·00 per cent. ...	6·00 per cent.
Cadbury's cocoa	1·82 ,, ...	7·15 ,,
Cocoa shell powder ...	8·98 ,, . .	not determined
,, ,, ...	8·03 ,, ...	11·95 ,,

The results of the analyses of the following samples of cocoa powders are given in full as the origin of the samples was not known :

Cold Water Soluble.	Total Nitrogen.	Fat.	Ash.	Alkalinity of Ash as K_2CO_3.	Pentosan.
Per cent.	Per cent.	Per cent.	Per cent.	Per cent.	Per cent.
18·60	3·23	28·82	8·18	4·15	2·18
18·08	3·15	29·74	8·12	4·08	2·31
18·56	3·06	28·57	8·38	3·79	2·35
18·08	3·29	28·24	9·03	3·79	1·69
18·08	3·20	28·21	8·84	3·75	1·84
18·48	3·24	27·51	9·30	4·08	1·89
19·00	3·32	28·19	8·61	4·08	2·08
17·44	3·07	26·82	7·18	2·77	2·81

These cocoas show no indications of added husk.

Of the several samples of chocolate powders given below, some were free from husk, whilst in the case of others the "cocoa" they contained consisted wholly of husk or cocoa-shell powder.

Cold Water Soluble.	Total Nitrogen.	Fat.	Ash.	Pentosan.	Fibre.
Per cent.	Per cent.	Per cent.	Per cent.	Per cent.	Per cent.
67·20	0·76	23·76	2·17	1·27	—
66·88	0·80	23·12	1·98	0·83	1·18
65·04	0·70	23·59	2·16	0·81	1·33
42·96	0·60	4·20	—	1·88	2·95
37·52	0·57	4·21	—	1·81	2·71

The two latter samples contained about 25 per cent. "cocoa" which was almost entirely husk.

We have had three samples of cocoa made up, the composition of which was not imparted to us until after the samples had been analysed and an opinion given on the same. The following were the analytical results:

Cold Water Soluble.	Total Nitrogen.	Fat.	Ash.	Pentosan.	Fibre.
Per cent.	Per cent.	Per cent.	Per cent.	Per cent.	Per cent.
31·11	1·14	21·93	1·66	1·39	3·01
41·90	0·72	6·07	2·90	1·55	1·67
15·88	3·09	31·91	4·66	2·45	7·01

From these figures the composition of the samples was calculated to be as follows:

	No. 1.	No. 2.	No. 3.
Cocoa	50	13	Contained about 5 per cent. husk, the rest being pressed cocoa powder.
Sugar	26	40	
Starch	24	34	
Husk	Not more than 1 per cent.	13	

The actual composition was:

	No. 1.	No. 2.	No. 3.
Cocoa	48	18	Contained 9 per cent. husk, the rest being pressed cocoa powder.
Sugar	24	38	
Starch	28	35	
Husk	None	9	

Feeding-Cakes.

	Moisture.	Oil.	Pentosan.	Fibre.
Linseed cake	11·90	8·08	3·38	6·76
Decorticated cotton-cake ...	7·70	10·20	7·22	5·02

	Pentosan.	Fibre.
Agar-agar	25·92 ...	0·28

Arrowroot (genuine St. Vincent).

Pentosan.
0·51 per cent.

In the case of mustard it will be seen that whilst the husk of mustard contains

a considerable quantity of furfuroid, and about an equal amount of crude fibre, the flour of mustard, which has been freed as completely as possible from husk, contains a very small quantity of pentosan, less than 1 per cent., whilst it does not appear to be possible to reduce the amount of fibre to less than 2 or 3 per cent. The determination of the pentosan, therefore, in the case of mustard, affords a surer guide for the estimation of the amount of husk it contains than does the determination of the crude fibre.

In pepper the pentosan estimation likewise appears to be a better guide in arriving at the percentage of husk than that of the fibre. While, in the two samples of pepper examined by us, the proportion of fibre was not quite twice as high in the black as compared with the white, the pentosan in the black was nearly three times that of the white.

In the case of cocoa, the advantage is still more on the side of the pentosan estimation. The best kinds of pressed cocoa contain as much as 6 to 7 per cent. of crude fibre, and cocoa-shell powder about 12 per cent.; while the amount of pentosan in pressed cocoa is only about 2 per cent., against 8 to 9 per cent. in the shell. While, therefore, the estimation of fibre is a very untrustworthy guide for the detection and determination of husk, the pentosans are capable of yielding a far more trustworthy indication.

How near it is possible to determine the relative proportions of cocoa, husk, sugar, and starch (the latter by difference), will be seen from the analyses of the three samples kindly made up for us by a well-known manufacturer.

The difference in the percentage of pentosan in coffee and chicory, although not very large, is yet sufficient to prove of assistance in the analysis of mixtures, whilst the determination of fibre quite fails, coffee and chicory in the roasted condition yielding similar quantities of fibre. During the roasting of coffee and chicory more or less of the substance is carbonized, and the carbon is estimated along with the crude fibre, whilst, as will be seen from the analyses of the five samples of chicory roasted for different lengths of time, the proportion of pentosan is not affected.

Discussion.

Mr. Bevan said that the method employed by Mr. Cross and himself was to determine furfural by precipitation with a solution of phenyl-hydrazine in acetic acid. The precipitate thus obtained was rather more than twice the weight of the furfural to be estimated, so that the error of manipulation was small.

ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

FOODS AND DRUGS ANALYSIS.

Estimation of the Nitrogenous Constituents of Malt, Wort, and Beer. B. V. Laszczynski. (*Zeit. f. a. gess. Brau.*, 1899, 140.)—The author finds that the albumin of wort and beer is completely precipitated by boiling for an hour under a pressure of one and a half atmospheres; that phosphotungstic acid and cupric hydroxide are not reliable reagents for separating nitrogenous bodies, since the former, though it precipitates the albumin, albumoses, and xanthine bodies completely, carries down an indefinite amount of the amide bodies; the latter precipitates the albumin completely, but also partly precipitates the albumoses and the amides. Krüger's process for the estimation of the xanthine bodies in animal fluids was found applicable to the determination of these bodies in wort and beer. He proposes the following scheme of analysis: The amount of water and of nitrogen are first determined in the sample of malt; 100 grammes of it are then finely powdered and mixed with 500 grammes of water in a tared beaker, and allowed to stand for two hours at the ordinary temperature under constant stirring with a machine. The mixture is then made up to its original weight, and filtered off. Of the filtrate, 25 c.c. are taken for the estimation of the nitrogen; this equals the total soluble nitrogen. Another 100 c.c. of the filtrate are placed in a flask and boiled in an autoclave for an hour at a pressure of one and a half atmospheres; the liquid is then filtered off hot, the coagulum washed with hot water, dried, and the nitrogen estimated in it; this equals the albumin nitrogen. The filtrate from this is evaporated down to 20 c.c., 2 drops of sulphuric acid added, saturated with zinc sulphate, and the albumose nitrogen estimated in the usual manner. The filtrate from the albumoses is heated to boiling with 10 c.c. of a saturated solution of sodium bisulphite, 10 c.c. of a 13 per cent. copper sulphate solution, and 5 c.c. of a 10 per cent. solution of barium chloride, allowed to cool, the precipitate filtered off, washed five times with hot water, dried, and the nitrogen it contains determined; this gives the nitrogen of the xanthine bodies. The amide nitrogen is calculated by deducting the nitrogen of these various bodies from the total soluble nitrogen. In the case of wort or beer 100 c.c. are taken directly. All the nitrogen determinations are made by the Kjeldahl process.

C. S.

Oil of Cognac. Schimmel and Co.'s April Report. (*Chem. Zeit.*, 1899, xxiii., 329.)—Various analytical data given by different specimens of oil of cognac are collected in the annexed table, from which it appears that although this product has hardly yet been sufficiently studied to enable its genuineness to be deduced from the constants, a low specific gravity and a high ester number point to the presence of the artificial oil. The first two samples from the Rhine and from Forst in Lusatia were specially prepared and purified for comparison with the others.

Description.	Sp. gr. at 15°.	Rotatory power at 20°.	Acid number.	Ester number.	Minimum strength and volume of alcohol to dissolve.
Rhenish oil, crude ...	0·878	−0° 3′	55·2	207·3	2 vol. of 80%.
,, refined ...	0·879	−0° 4′	50·9	218·6	,, ,,
Lusatian oil, crude ...	0·880	−0° 10′	70·9	210·8	,, ,,
,, refined ...	0·880	−0° 11′	68·6	140·9	,, ,,
Spanish oil	0·876	—	37·1	212·4	insoluble in 80%, 0·5 vol. of 90%.
Commercial oil ...	0·883	+0° 43′	105·5	172·8	2 vol. of 80%.
Artificial oil	0·871	±0°	5·8	291·7	10 vol. of 70%.

F. H. L.

ORGANIC ANALYSIS.

The Characteristics of Oleo-distearin. R. Henriques and H. Kühne. (*Berichte*, 1899, xxxii., 387-394.)—Heise (*Arbeit. a. d. Kaiserl. Gesundheitsamt.*, 1896, 540) found that Mkani fat, which is derived from the seeds of the East African tallow-tree (*Stearodendron Stuhlmanni*), consisted essentially of a mixed glyceride containing two stearic acid radicles and one oleic acid radicle. As doubt has since been thrown on the correctness of Heise's conclusions, the authors have made a number of experiments on the subject.

By crystallizing a large quantity of the fat five times from a mixture of ether and alcohol they obtained a substance melting at 44·5° to 45·5° C., but which, having once been melted, had a melting-point of 38·5° to 39·5° C. As no further alteration could be brought about by continued crystallizations from absolute alcohol and from glacial acetic acid, the conclusion was arrived at that Heise's oleo-distearin was a definite chemical substance notwithstanding the fluctuations in its melting-point. Its iodine value was 28·6, and its molecular equivalent 295·2, the theoretical values for oleo-distearin being 28·6 and 296·0 respectively.

On treating the glyceride with Waller's iodine solution, or by dissolving it in ether and adding an alcoholic solution of iodine chloride, the chlor-iodo addition compound crystallized out almost quantitatively. This had the same appearance and almost the same solubility in various solvents as the oleo-distearin. It melted at 44·5° to 45·5° C., and after having once been melted and again solidified, had a melting-point of 41·5° to 42·5° C. It was very stable, and retained about two-thirds of its chlorine after repeated boiling with alcoholic potassium hydroxide. Boiling with quinoline or aniline, however, was found to remove the whole of the halogens.

Oleo-distearin yielded an elaidin, but with less readiness than olein. This had only one melting-point (61° C.). It readily formed a chloro-iodide, which melted at 57° to 58° C., and, like the elaido-distearin, did not change its melting-point after fusion.

The authors consider that it may be possible to isolate similar mixed glycerides from other fats and oils in the form of their chloro-iodides, and have already obtained crystalline compounds in this way from butter and from linseed oil, which appear to have properties similar to the substance described above (*cf.* Hehner and Mitchell, ANALYST, xxiii., 317). C. A. M.

On the Saponification Value of Fish-Oils. W. Fahrion. (*Chem. Rev. Fett-u. Harz-Ind.*, vol. vi., pp. 25-29.)—The following values were obtained by the author in a new series of investigations:

	Iodine Value.	Henriques' Cold Saponification Method.		Hot Saponification Method. Henriques' Reagent.	Saponification according to Becker.	Inner Saponification Value by the Fahrion Method.
		24 hours.	2-8 days.			
Sardine-oil	191·7	189·5-195·9	192·5-216·8	190·9-193·8	196·2-200·3	185·2-186·4
Stickleback-oil ...	162·0	183·2-190·7	184·5-209·9	186·6-189·9	190·6-192·6	181·1-181·5
Seal-oil	146·2	186·0-190·4	186·7-197·4	190·7-192·9	189·8-193·5	184·4-186·0
Haddock-oil	166·2	182·3-183·3	183·5-185·5	182·5-183·0	187·8-188·3	181·6-181·9
Japan fish-oil	108·5	186·0-186·8	186·1-189·2	186·6-186·8	189·3-191·4	180·0-180·9
Whale-oil	106·1	184·0-185·1	184·5-188·0	184·5-185·5	191·2-193·5	174·8-175·7
Linseed-oil	172·0	187·0-187·7	187·0-188·6	187·6-189·1	188·8-192·3	186·8-187·9

The abnormally high values furnished by the first three oils in the longer period cold method are due to the formation of low molecular volatile fatty acids under the prolonged influence of alkali on these oils. That oxy-fatty acids, insoluble in petroleum spirit, are also formed has now been ascertained by the author, who obtained from 10 grammes of sardine-oil 0·0413 gramme of liquid and 0·0079 gramme of solid oxy-acids, together with 0·0448 gramme of volatile fatty acids (m.w. 73·9), and also succeeded in isolating oxy-acids from saponified stickleback-oil; and it is to this partial oxidation of the fatty acids that he ascribes the irregularities in the saponification value of these fish-oils, though the temperature of the reaction directly affects the results. The conditions causing these irregularities disappear for the most part when the test is performed without any excess of alkali, as is the case in determining the inner saponification value. The evaporation of the fatty-acid solution in petroleum spirit or alcohol should, however, be effected on the water-bath, and not in a drying-oven, at 100° C., since under the latter conditions the acids undergo a modification which falsifies the results.

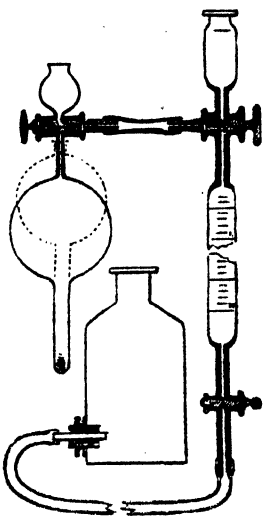
In the hot method of saponification the proportion of water in the alkali has an influence on the results, Becker's reagent, with about 16 per cent. of water, giving higher values and requiring longer time to react than that of Henriques, which contains only 3 to 4 per cent. The author recommends the latter, as giving results more in accordance with the cold method, and he agrees with Henriques as to the advisability of dissolving the alkali direct in 96 per cent. alcohol in place of the usual procedure. C. S.

On the Reduction of Chromic Acid by Acetic Acid, and its Effect in Anthracene Testing. H. Bassett. (*Chem. News*, vol. lxxix., p. 157.)—Owing to certain irregularities in the results obtained during a series of experimental tests on anthracene, the acetic acid employed (crystallizing at 56° F.) was subjected to examination and compared with the best acid, crystallizing at 62° F., the solutions being prepared exactly as in the ordinary test for anthracene (with chromic acid of 98 per cent.). It was found that after twenty-four days the loss of chromic acid by reduction amounted to 0·87 gramme (out of 15 grammes taken) in the case of the

inferior acetic acid, and to 0·16 gramme in the other, owing to the presence of aldehyde, acetone, etc., in the former. Under the usual conditions of the anthracene test, a blank experiment gave, after four hours' boiling, a loss of 6·45 and 4·44 grammes respectively. The reduction, however, fell to almost *nil* when equal volumes of acid and water were used.

The use of inferior acid leads to results higher than the truth, the difference in the present instance being equal to about 0·4 per cent. of anthracene; and a similar error (of about 0·3 per cent.) arises from the use of a reagent that has been stored for about a month. It is therefore advisable to employ pure acetic acid, and an oxidizing mixture not more than fifteen days' old. C. S.

Analysis of Illuminating Gas. O. Pfeiffer. (*J. Gasbeleucht.*, 1899, xlii., 209; through *Chem. Zeit. Rep.*, 1899, 129.)—The author has abandoned absorption methods except for the carbon dioxide and the heavy hydrocarbons; he prefers processes of gradual combustion. His absorption apparatus is figured herewith; and for the explosions he uses a burette, which holds 110 c.c., fitted with stoppers at both ends. After explosion and removal of carbon dioxide, the excess of oxygen is absorbed with phosphorus, thus leaving the nitrogen of the original sample. A complete analysis of coal-gas can be carried out in forty-five minutes, the formulæ employed being the same as those quoted by Dennis and Hopkins (ANALYST, vol. xxiv., p. 106, Nos. 7, 8, and 9). F. H. L.



The Use of Silico-tungstic Acid as a Reagent for Alkaloids. G. Bertrand. (*Bull. Soc. Chim.*, 1899, xxi., 434-439.) — Silicotungstic acid ($12WO_3.SiO_2.2H_2O$) and its alkaline salts give precipitates with alkaloids in sufficiently concentrated solution, which can be readily collected on a filter. The precipitates are white, pale yellow, buff, or salmon coloured. They are insoluble, or nearly so, in cold water, but are somewhat soluble in boiling water. When dried at 30° C. to constant weight they retain a certain proportion of their combined water, the amount varying with the alkaloid A further part, at least, of this water is liberated at 120° C.

These neutral alkaloidal silicotungstates generally contain four equivalents of alkaloid to one of silico-tungstic acid:

$$12WO_3.SiO_2.2H_2O.4Alk + nH_2O.$$

They resist the action of concentrated mineral and organic acids, and on calcination leave a fixed residue of silicic and tungstic acids.

The pyridine salt ($12WO_3.SiO_2.2H_2O.4C_5H_5N + H_2O$) is a white crystalline powder, which becomes anhydrous at 120° C. It is slightly soluble in boiling water, from which it crystallizes on cooling in small colourless prisms.

Morphine silicotungstate ($12WO_3.SiO_2.2H_2O.4C_{17}H_{19}NO_3+9H_2O$) forms an amorphous salmon-coloured mass, which retains two molecules of water at 120° C.

The strychnine salt ($12WO_3.SiO_2.2H_2O.4C_{21}H_{22}N_2O_2+8H_2O$) resembles the morphine compound, but is of a buff colour, and only retains one molecule of water at 120° C.

On heating an alkaloidal silicotungstate in the liquid in which it was precipitated, it immediately loses a portion of its water. This body is also obtained in the form of a fine precipitate, which is not readily filtered, by adding the reagent to a hot solution of the alkaloid.

The silicotungstates of morphine and strychnine thus obtained have the following composition when dried at 30° C.:

Morphine silicotungstate ∴ $12WO_3.SiO_2\ 2H_2O\ 4C_{17}H_{19}NO_3+8H_2O$.
Strychnine silicotungstate ... $12WO_3.SiO_2.2H_2O.4C_{21}H_{22}N_2O_2+7H_2O$.

When testing a solution qualitatively for an alkaloid, it is preferable to attempt to form the salts containing the smaller amount of water, since these are often more perceptible than the salts precipitated from cold solutions. Thus, if 1 or 2 drops of the reagent be added to 5 c.c. of a solution of aconitine sulphate (1 : 50000), there is only a very slight opalescence; but on warming the liquid to the boiling-point, it becomes clear, and then on cooling a fine powdery precipitate appears. This difference is still more pronounced in the case of veratrine, it being possible to detect one part in 130,000 by warming the solution after the addition of the reagent.

The author has determined the limits of sensibility of this reaction with the principal alkaloids, and finds that they can be classified into two groups, viz., those whose silicotungstates are equally perceptible in hot and cold liquids, and those whose silicotungstates become more visible on warming the solution to 100° C. and then cooling.

I.	One part in :	II.	One part in :
Pyridine	3,000	Aconitine	80,000
Coniine	8,000	Veratrine	130,000
Morphine	16,000	Brucine	150,000
Theobromine	18,000	Strychnine, Narcotine	200,000
Nicotine	20,000	Quinine, Quinidine, Cinchonine, Cinchonidine ...	500,000
Quinoline, Narceine	30,000		
Codeine	40,000		
Atropine, Caffeine	50,000		
Cocaine	200,000		

Caffeine and theobromine are exceptional in requiring the addition of a sufficient quantity of free acid (*e.g.*, 3 to 4 per cent. of hydrochloric acid) if their full limit of sensibility to the reaction is to be reached. The caffeine silicotungstate formed under these conditions has the formula:

$$12WO_3.SiO_2.2H_2O.3C_8H_{10}N_4O_2+6H_2O.$$

Notwithstanding their stability, the silicotungstates of alkaloids are attacked by

certain reagents, noticeably by oxidizing bodies. It is thus possible in certain cases to obtain the characteristic colour reaction of the alkaloid directly with the precipitate—as, for example, in the case of strychnine with sulphuric acid and bichromate, and of morphine with Fröhde's reagent.

The alkaloids can be recovered from the precipitates by treating the latter with an alkali, which instantly decomposes them, and the alkaloid can then be extracted from the liquid by the usual methods.

The author has also made experiments to determine the action of silicotungstic acid on a number of glucosides, such as arbutin, coniferin, digitalin, ouabain, salicin, etc.; on certain bitter principles, such as picrotoxin, santonin, and quassin; on amides, such as urea, asparagine, leucine, and tyrosine; and on tannin, gum arabic, and other bodies which are often found accompanying alkaloids in plants. With the exception of albuminous substances, however, no precipitate was obtained with any of them, either in neutral or in acid solution. C. A. M.

A Modification of Denigés Method of Estimating Uric Acid. (*Ann. de Chim. Anal.*, 1899, iv., 82, 83.)—The method described below combines the methods of Denigés and of Hopkins. The phosphates are first removed by adding to 100 c.c. of the urine 10 c.c. of a solution of 160 grammes of anhydrous sodium carbonate in a litre of water; 82 c.c. of the filtrate are taken, and the uric acid rendered insoluble either by the addition of 30 c.c. of a mixture of 10 c.c. of Fehling's solution with 20 c.c. of sodium bisulphite solution (36° to 40° Bé), as proposed by Kluger; or, as described by Denigés, by the addition of 5 c.c. of a solution of copper sulphate (40 grammes per litre) and 20 c.c. of a solution containing sodium thiosulphate, 100 grammes, and sodium potassium tartrate 100 grammes, in a litre of water.

The mixture is shaken and filtered, and the filter washed with water until free from alkali, and thrown into 500 c.c. of water acidified with 5 c.c. of sulphuric acid. After the liquid has been well shaken for several minutes in contact with the air, decinormal permanganate solution is added drop by drop until a permanent pink tint is obtained.

If n c.c. be used in the titration, the amount of uric acid in a litre of the urine can be calculated by the formula,

$$\frac{n \times 0{\cdot}00745 \times 1000 \times 110}{82 \times 1000} = n \text{ decigrammes};$$

or, in other words, the number of c.c. used gives directly the decigrammes of uric acid.

The relation between decinormal permanganate and uric acid (1 c.c. = 0·00745 gramme) was obtained experimentally with a specimen of that acid purified by means of sulphuric acid. C. A. M.

Note on Kjeldahl's Method of Determining Nitrogen. Maquenne and Roux. (*Bull. Soc. Chim.*, 1899, xxi., 312-314.)—Referring to the precipitation of mercury by means of sodium sulphide, the authors point out that there are two main

objections to this course. Firstly, volatile sulphur compounds are formed, which distil with the ammonia, and interfere with the sharpness of the subsequent titration, especially when turmeric is used as indicator; and, secondly, the mercuric sulphide formed is not completely insoluble in alkaline sulphides, and there is nothing to prevent the formation of compounds of ammonia and mercury, which are not readily decomposed by alkalies, with the result that the yield of ammonia may be too low, as is shown by the figures in the subjoined table.

These drawbacks are obviated by precipitating the mercury from the acid solution by means of sodium hypophosphite. About 1 gramme of this salt is introduced into the acid solution while still warm, the whole heated to 60° to 70° C., then cooled, sodium hydroxide added, and the ammonia distilled in the usual manner.

The comparative results obtained by this modification, by the ordinary method, and by the soda-lime method were:

Material.	Kjeldahl's Method.		Soda-lime Method.
	Precipitation with Hypophosphite.	Precipitation with Sulphide.	
Cotton-seed cake	4·45	4·39	4·48
Colza cake	6·84	6·70	6·87
Wheat flour	1·86	1·89	1·91
Bean flour	5·24; 5·25	—	5·29
Powdered horn	14·31; 14·28	14·12; 14·11	14·30
Dried blood	7·73; 7·78	7·69; 7·79	7·78

C. A. M.

INORGANIC ANALYSIS.

The Colorimetric Estimation of Nickel. M. Lucas. (*Bull. Soc. Chim.*, 1899, xxi., 432, 433.)—The red colour which salts of nickel give with potassium or ammonium tri-thiocarbonate is proportional to the amount of nickel, and can be used for the colorimetrical estimation of that metal.

Ammonium tri-thiocarbonate is prepared by digesting freshly-prepared ammonium sulphide at a moderate heat for twenty-four hours with one-twentieth part of its weight of carbon bisulphide.

Potassium tri-thiocarbonate may be obtained by saturating one-half of a 5 per cent. solution of potassium hydroxide with hydrogen sulphide, adding the other half, digesting the whole at a moderate heat with a twenty-fifth of its volume of carbon bisulphide, and separating the dark-reddish liquid from the unaltered carbon bisulphide.

The darker-coloured of the two reagents is diluted with water until of the same colour as the other. Both are kept in well-stoppered bottles.

In order to obtain uniform and durable colorations with nickel, it is necessary to add a large excess of the reagents. But since, as a rule, the quantity of nickel to be estimated is less than 2 milligrammes in 100 c.c., the colour of the reagents themselves does not introduce a serious error. The colour produced by 10 c.c. of one of these in 100 c.c. corresponds to not more than one-tenth of a milligramme of the

metal, and, by using typical colour standards, the drawback becomes one of diminution of sensibility.

Cobalt salts give a brown coloration under the same conditions, so that it is necessary to separate any cobalt present before determining the nickel. Ammonium tri-thiocarbonate gives a much more pronounced colour with cobalt than does potassium tri-thiocarbonate—a property which may be used as a test for the presence of that metal.

Copper, too, gives a colour almost as pronounced as that of nickel. When present, it may be determined colorimetrically by means of potassium ferrocyanide and the amount deducted from the combined nickel and cobalt as estimated with tri-thiocarbonate, but it is preferable to remove it previously.

In analysing a steel containing nickel, 0·5 gramme of the sample is dissolved in *aqua regia*, and two successive precipitations made with ammonium hydroxide and ammonium chloride. The filtrate is diluted to 500 c.c., and three portions of 50 c.c. each taken. To the first is added 10 c.c. of the solution of ammonium tri-thiocarbonate, and to the second the same volume of the solution of potassium tri-thiocarbonate. If the coloration is a darker brown in the first than in the second, the presence of cobalt with the nickel is indicated, and a separation is made by one of the usual methods.

If not, the first of the test-tubes is compared with a standard colour-scale consisting of tubes containing the same volume of liquid and approximately the same proportion of ammonium salts, with gradually increasing quantities of nickel. These must be prepared with boiled water, and kept free from contact with the air. The tri-thiocarbonate is only added to them after the liquid has been diluted to the required volume.

The third portion of the ammoniacal filtrate is used for a corroborative determination.

Finally, another portion is tested with potassium ferrocyanide for copper, after evaporation of the ammonia. C. A. M.

Separation of Nickel and Zinc. E. Döhler. (*Chem. Zeit.*, 1899, xxiii., 399.)—The acid solution of the two metals, containing about 0·05 gramme of zinc, is evaporated nearly to dryness, diluted with water, neutralized with ammonia, and mixed with 0·2 gramme of sodium formate and 5 c.c. of formic acid. The liquid is raised to 60° C., and treated with a current of sulphuretted hydrogen for twenty minutes. The zinc sulphide is collected on a double paper, washed with sulphuretted hydrogen water, and either dissolved at once in hydrochloric acid, or dried, ignited in porcelain, and dissolved. The solution is evaporated till most of the acid is removed, diluted, freed from traces of iron, etc., with ammonia, and in the filtrate the zinc is titrated with sodium sulphide. For very exact work the zinc sulphide should be thrown down with sulphuretted hydrogen a second time, and estimated gravimetrically by ignition in a stream of hydrogen, as the volumetric process yields results slightly below the theoretical. Instead of the formic acid and formate, 1 gramme of sodium acetate and 40 c.c. of "acetic acid" may be employed, but the former method is preferable. F. H. L.

Estimation of Potash. E. W. Bell. (*Chem. News*, vol. lxxix., p. 135.)—To shorten the time required for a potash estimation, and to obtain a purer precipitate than by the ordinary method, the author recommends, in the case of *manures*, to well boil 5 to 10 grammes of the sample with about 150 c.c. of water, with or without a little hydrochloric acid; then render slightly alkaline with ammonia and add barium carbonate in large excess (about double the weight of the substance taken). After boiling for half an hour longer, the precipitate is filtered off, washed, and the filtrate and washings are made up to 500 c.c.

Fifty to 100 c.c. of the solution are evaporated to dryness with a small quantity of ammonium oxalate, gently ignited, treated with hot water, and filtered, the filtrate being evaporated with hydrochloric acid and platinum chloride and finished as usual.

In the case of *soils* (if the potash soluble in hydrochloric acid is required) the hydrochloric solution is rendered alkaline with ammonia, treated with barium carbonate, etc., as above, or the soil is moistened with strong sulphuric acid, gently ignited, and boiled with water, the barium carbonate precipitation and final treatment being as before, except that the ammonium oxalate and ignition are dispensed with.

For estimations in *vegetable substances*, these must be moistened with sulphuric acid, ignited, and treated in the same way as soils. C. S.

The Constitution of the Ammonium-Magnesium Phosphate of Analysis. F. A. Gooch and M. Austin. (*Zeit. für Anorgan. Chemie*, xx., 121)—The authors have carried out a series of experiments upon the precipitation of ammonium-magnesium phosphate under the various conditions that might occur in analysis. A few experiments were made to determine to what extent ammonium chloride might exert a solvent action upon the precipitate in presence of the precipitant. It was found that as little as 0·001 gramme of magnesium oxide, in the form of nitrate, in 500 c.c. of faintly ammoniacal water, could be detected by the opalescence produced on adding 1·75 gramme of microcosmic salt, even when 60 grammes of ammonium chloride were present. The authors consider that strongly ammoniacal fluids are unnecessary in the precipitation of ammonium-magnesium phosphate, and in nearly all the experiments mentioned the solutions and wash-water were made only faintly ammoniacal. In a series of experiments made by precipitating magnesium nitrate with considerable excess of microcosmic salt in the presence of varying amounts of ammonium chloride, the results showed, in all cases, errors of excess, the greatest occurring where the largest amounts of ammonium chloride had been used. When, however, the supernatant liquid was filtered from the precipitate by decantation, and the latter was redissolved in hydrochloric acid and reprecipitated with a faint excess of dilute ammonia, without any addition of ammonium chloride, the error disappeared altogether, and good results were obtained. If the precaution of redissolving be taken, the quantity of ammonium chloride originally present does not affect the final results, unless, indeed, the amount is so large that an appreciable quantity is left even after redissolving. The influence of free ammonia during the precipitation was also studied. The results indicate a tendency for free ammonia,

ammonium chloride, and excess of phosphoric acid to produce a salt containing a larger proportion of ammonia and a smaller proportion of magnesium than are contained in normal ammonium-magnesium phosphate. For the determination of phosphoric acid the authors recommend the observance of the following conditions: The phosphate solution to contain not more than 5 to 10 per cent. of ammonium chloride; the magnesia mixture to be only in slight excess; the ammonia, both for precipitation and washing, to be used sparingly; the precipitate to be redissolved and reprecipitated in cases where too much ammonium chloride, or double the theoretical quantity of magnesia mixture, has been used. H. H. B. S.

Determination of Sulphur in Iron Pyrites. O. N. Heidenreich. (*Zeit. für anorgan. Chemie*, xx., 233.)—When sulphuric acid is precipitated by barium chloride in the presence of a considerable quantity of iron, the barium sulphate is found to be contaminated with ferric sulphate (Jannasch and Richards, *Jour. pr. Chemie*, xxxix., 321-334). The author suggests that this drawback may be overcome by reducing the ferric salt by means of zinc before the addition of the barium chloride. This is only a preliminary note on the subject; but the author gives the results of three determinations made in this way with different quantities of the same sample of pyrites, which agree well, and he further mentions that the barium sulphate obtained in each case was almost white. H. H. B. S.

On the Influence of Hydrochloric Acid in Titrating with Sodium Thiosulphate, with Special Reference to the Determination of Selenious Acid. J. T. Norton. (*Zeit. für anorgan. Chemie*, xx., 221-229.)—It appears from the author's experiments that the influence of hydrochloric acid upon the thiosulphate depends primarily upon the quantity of thiosulphate present, and then upon the degree of dilution and the quantity of the hydrochloric acid. In titrating large quantities of thiosulphate with iodine in presence of hydrochloric acid, the temperature should be maintained at as nearly 0° C. as possible, and the titration should be carried out quickly. So long as the quantity of thiosulphate does not exceed 20 c.c. of a $\frac{N}{10}$ solution, the titration in cold solutions, varying in bulk from 100 to 500 c.c. at the commencement of the titration, proceeds pretty regularly, even when 10 c.c. of hydrochloric acid are present. When, however, the quantity of thiosulphate exceeds this limit, it is necessary to diminish the acid and to work with a solution having a bulk of 400 to 500 c.c.

In most cases in which this process is made use of the thiosulphate is run into the solution to be tested, and, since decomposition takes place at the moment when the thiosulphate meets the solution, hydrochloric acid ceases to have any influence. In Norris and Fay's method for the iodometric determination of selenious acid, however, an excess of thiosulphate is added simultaneously with hydrochloric acid, so that conditions prevail which render necessary the careful regulation of the quantity of acid and thiosulphate and the degree of dilution.

Norris and Fay's method is briefly as follows: A solution of selenious acid in ice-cold water is treated with an excess of a $\frac{N}{10}$ solution of thiosulphate in the

presence of hydrochloric acid, and the excess of thiosulphate is then titrated back with iodine.

The author has made experiments to determine the exact conditions which it is necessary to observe in order to obtain accurate results by this method. The most important point appears to be the restriction of the excess of thiosulphate within narrow limits, which is not a difficult matter if the approximate percentage of selenious acid be known. The conditions should then be so arranged that never more than 20 c.c. of $\frac{N}{10}$ thiosulphate are used beyond the quantity required for the reduction of the selenious acid. If this be done, and the solution be diluted to 400 c.c., 10 c.c. of hydrochloric acid may be used without adversely affecting the results. If, however, the quantity of hydrochloric acid added be restricted to 5 c.c., which, in the author's opinion, is sufficient, the solution need not be diluted beyond 200 c.c. H. H. B. S.

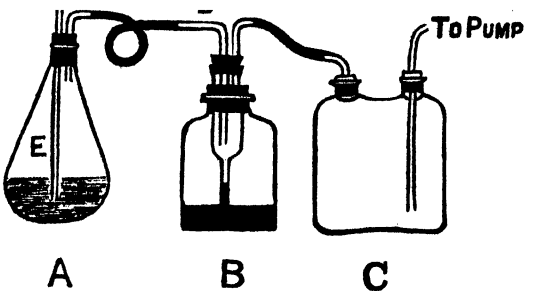
A Study of the Relative Value of Lacmoid, Phenacetolin, and Erythrosine as Indicators in the Determination of the Hardness of Water by Hehner's Method. J. W. Ellms. (*Jour. Amer. Chem. Soc.*, xxi., 359-369.)—The principal conclusions arrived at are as follows: There is very little to choose between the three indicators, and with proper care and skill, and with a knowledge of the true end-point with lacmoid and phenacetolin, all may be said to be suitable for the purpose. Erythrosine, however, gives more uniform results and a larger percentage of the carbonates with low than with high amounts, whilst the reverse seems to be true of lacmoid and phenacetolin. If, therefore, uniformity of results within very narrow limits is desired, the preference should be given to erythrosine. Erythrosine may also be used with fairly turbid and coloured waters, and in this respect is preferable to either lacmoid or phenacetolin, which have a tendency to give too high results with such waters. H. H. B. S.

A Delicate Reaction of Hydrogen Peroxide. E. S. Barralet. (*Chem. News*, vol. lxxix., p. 136.)—The author proposes to utilize the colour change produced by the oxidizing action of hydrogen peroxide on ferrous ferrocyanide as a means of detecting the former when present in very minute quantities, 1 c.c. of a $\frac{1}{1\,85000}$ solution of hydrogen peroxide being sufficient to intensify the pale blue colour of ferrous ferrocyanide in 20 c.c. of the freshly-precipitated compound. The reagent should be freshly prepared for each test, or else be stored in a bottle under a layer of petroleum. C. S.

APPARATUS.

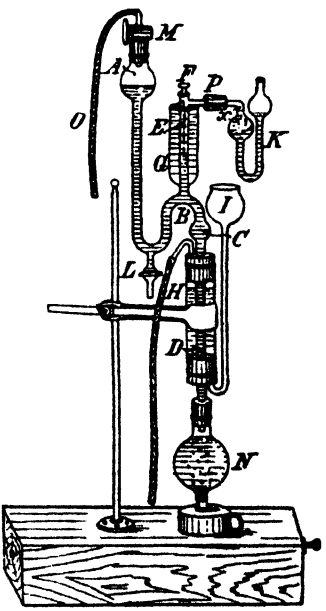
A Filter-Pump Accessory. H. Jervis. (*Chem. News*, vol. lxxix., p. 86.)—To prevent the reversal of current due to back pressure, the author recommends the arrangement shown in the drawing, in which C is the reservoir bottle, A the dissolving flask, and B the safety appliance. In this latter the drawn-out tube is held firmly in the neck of the bottle and dips just below the surface of the mercury. Owing to the narrowness of the tube, considerable pressure is required to lower the level of the mercury, and hence no air can pass; but should the water run back into

C, the compressed air forces the mercury below the bottom of the drawn-out tube, and escapes before the liquid in A is pushed beyond any given point in the vertical tube E. To render the degree of exhaustion constant, mercury is poured into B until it reaches nearly to the bottom of the larger tube D, the end of this tube becoming covered by the mercury as soon as the pump is started.



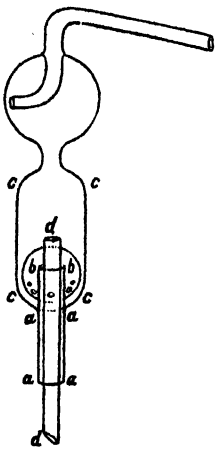
C. S.

Apparatus for Measuring the Gases dissolved in Water. F. C. G. Müller. (*Zeits. angew. Chem.*, 1899, 253.)—In the accompanying illustration this apparatus is drawn to a scale of 1 : 5. The flask *N* holds 100 c.c., and is completely filled with the sample, which has been made slightly alkaline. It is put in position, the stopcock *F* is withdrawn, and the whole pipette is charged with petroleum through *A* until the oil reaches the capillary *P*. *F* is then inserted and closed as against *K*, and the water is boiled for ten minutes to liberate the gases which collect in the graduated tube *E*, while the paraffin is driven back into *A*. The ebullition is conducted fast enough to fill *D* with steam, but not with sufficient rapidity to force the oil upwards out of *C*. The level of the petroleum is next adjusted by means of *L*, and the volume of gas read off, any bubbles in the burette being broken by sharply aspirating through *O*. *F* is then turned through 180°, and the oxygen is absorbed by forcing the gas into *K* several times, finally drawing it back into *E* to measure the residual nitrogen. Corrections for temperature, etc., are made by the help of a gas-volumeter. At the end of one test, by producing a partial vacuum in *A*, the oil can be kept from escaping while a fresh flask is put into position ; but it is advisable to wipe *E* out, which can be done through *F* after the petroleum has been drawn back into *A*. The apparatus is compact, and can be fitted into a portable case with a few spare flasks, so that with a second case holding standard alkali, phenolphthalein, a burette, and other apparatus for titrating the carbon dioxide, a complete analysis of the gaseous constituents of water can easily be carried out on the spot.



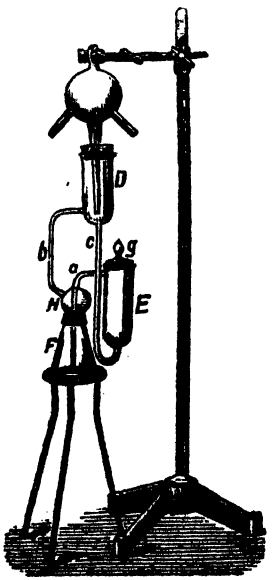
F. H. L.

Gas Washing Apparatus. O. Foerster. (*Chem. Zeit.*, 1899, xxiii., 196.)—This apparatus is specially designed for use during the estimation of nitrogen in



saltpetre by Kühn's process, its function being to return the condensible vapours to the flask in order to avoid the necessity for stopping the operation at intervals, lest the solution become too concentrated, and simultaneously to wash the permanent gases in the condensed liquid. The vapours leave the vessel through the annular space *a*, pass into the bulb *b*, and escape through the holes; the steam condenses in *c*, and gradually fills the lower part thereof until it reaches the level of *d*, when the surplus flows back regularly into the flask through the innermost tube. Thus, the vapours issuing from the various holes in *b* have to bubble through a column of the previously-condensed liquid. By bending the lower extremities of *a* and *d*, the former can be joined to the upper part of a hollow stopper which also carries a funnel and stopcock; the stopper itself fits the reaction flask, so that the whole arrangement may be used for charging as well as distilling off the contents of the vessel underneath. As a lubricant, a mixture of equal parts of ceresin and vaselin is recommended. F. H. L.

Apparatus for the Extraction of Liquids with Ether. F. Baum. (*Chem. Zeit.*, 1899, xxiii., 249.)—This apparatus has, *inter alia*, the advantage that it serves, without alteration or rearrangement of its parts, for the extraction of a solution by means of an immiscible liquid, and also for the distillation of the solvent from the extract. *H* is a hollow glass cap ground to fit on the outside of the neck of *F*. The vapours rise through *b*, condense in *D* and the condenser above, descend through *c*, pass through the material in *E*, and return viâ *a*. A small lateral hole is drilled in the stopper *g*, which can be placed in connection with a similar aperture in the side of *E*, thus bringing the interior to atmospheric pressure. The whole apparatus is slung from the condenser by the cork in *D*. *F* is also fitted with a plain glass cap, to be used during the weighing of the extract. *E* is filled almost up to the level of *a* with the aqueous liquid, and *F* is immersed in a water-bath; the holes in *gE* are in juxtaposition. When the operation is finished, the aqueous liquor is pipetted out of *E*; and as the ether continues to boil, it collects therein until *F* is practically free from solvent and merely requires to be dried. It is convenient to have several flasks like *F* all fitting the same stopper; if this be done, a second extraction can be started immediately. The new flask is placed in position, the fresh aqueous solution poured into *E* (which is, as just mentioned, full of recovered solvent); the ether is then displaced, runs into *F*, and the whole is ready once more. By the employment of a paper cone, etc., solids can be treated in *E* similarly. F. H. L.



ERRATUM.—Page 145, four lines from bottom, *for* "one-eighth of a gramme," *read* "0·8 gramme."

THE ANALYST.

AUGUST, 1899.

PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS.

THE COMPOSITION OF MILK AND MILK PRODUCTS.

By H. Droop Richmond.

(*Read at the Meeting, April* 12, 1899.)

Of the 36,399 samples analysed in the Aylesbury Dairy Company's laboratory during 1898, 29,707 were samples of milk; the average composition of 14,135 taken from the railway churns on arrival of the milk from the farms is given in Table I.; the composition of the morning and evening milk is given separately.

Table I.

Average Composition of Milk Inwards during 1898.

Month.	Morning Milk.				Evening Milk.				Average.			
	Specific Gravity.	Total Solids.	Fat.	Solids-not-Fat.	Specific Gravity.	Total Solids.	Fat.	Solids-not-Fat.	Specific Gravity.	Total Solids.	Fat.	Solids-not-Fat.
January	1·0322	12·55	3·64	8·91	1·0320	13·05	4·11	8·94	1·0321	12·80	3·87	8·93
February	1·0321	12·55	3·66	8·89	1·0318	13·03	4·12	8·91	1·0320	12·79	3·89	8·90
March ...	1·0323	12·58	3·64	8·94	1·0319	13·03	4·10	8·93	1·0321	12·80	3·87	8·93
April ...	1·0323	12·56	3·63	8·93	1·0321	13·07	4·10	8·97	1·0322	12·82	3·87	8·95
May ...	1·0326	12·43	3·46	8·97	1·0323	13·05	4·03	9·02	1·0324	12·74	3·74	9·00
June ...	1·0325	12·28	3·36	8·92	1·0322	12·70	3·76	8·94	1·0323	12·49	3·56	8·93
July ...	1·0320	12·37	3·54	8·83	1·0316	12·63	3·84	8·79	1·0318	12·50	3·69	8·81
August ...	1·0315	12·30	3·57	8·73	1·0312	12·72	3·99	8·73	1·0314	12·51	3·78	8·73
September	1·0317	12·48	3·68	8·80	1·0312	12·79	4·04	8·75	1·0315	12·63	3·86	8·77
October...	1·0321	12·73	3·81	8·92	1·0317	13·05	4·15	8·90	1·0319	12·89	3·98	8·91
November	1·0323	12·76	3·80	8·96	1·0319	13·11	4·16	8·95	1·0321	12·94	3·98	8·96
December	1·0324	12·72	3·73	8·99	1·0322	13·08	4·07	9·01	1·0323	12·90	3·90	9·00
Average...	1·0322	12·53	3·63	8·90	1·0318	12·94	4·04	8·90	1·0320	12·73	3·83	8·90

The mean composition does not differ appreciably from that found last year, the fat being slightly higher and the solids-not-fat slightly lower. As has been invariably noticed in former years, the solids-not-fat in July begin to fall, and attain a minimum in August. Usually a rapid rise in solids-not-fat takes place in September, but in 1898 the solids-not-fat remained low during this month, and did not rise till October. In the respect of low solids-not-fat, 1898 resembled 1893, and it may be remarked that both years were seasons of drought in the summer.

Fifty analyses have been made during the year, in which more figures have been determined than is usual in an ordinary milk analysis. They are tabulated below (Tables II., III., and IV.):

TABLE II.

Normal Milk (Morning Meal).

Specific Gravity.	Total Solids.	Fat.	Sugar.	Proteids.	Ash.	Solids-not-Fat.
1·0330	12·15	3·30	4·77	3·33	0·75	8·85
1·0320	12·31	3·60	4·70	3·27	0·74	8·71
1·0325	12·41	3·35	4·70	3·60	0·76	9·06
1·0325	12·73	3·85	4·82	3·32	0·74	8·88
1·0323	11·79	3·05	4·81	3·19	0·74	8·74
1·0330	12·24	3·30	4·84	3·37	0·73	8·94
1·0332	12·28	3·15	4·92	3·48	0·73*	9·13
1·0326	12·85	3·85	4·63	3·61	0·76	9·00
1·0326	11·64	2·95	4·58	3·40	0·71	8·69
1·0327	12·12	3·35	4·75	3·29	0·73	8·77
1·0328	12·80	3·95	4·82	3·30	0·73	8·85
1·0342	12·64	3·50	4·86	3·53	0·75	9·14
1·0330	12·38	3·40	4·66	3·56	0·76	8·98
1·0320	12·36	3·53	4·66	3·42	0·75	8·83
1·0329	12·79	3·70	4·60	3·76	0·73	9·09
1·0325	12·10	3·25	4·68	3·43	0·74	8·85
1·0327	12·26	3·35	4·88	3·29	0·74	8·91
1·0315	12·60	3·75	4·56	3·56	0·73	8·85
1·0315	11·86	3·15	4·50	3·48	0·73	8·71
1·0320	11·98	3·35	4·58	3·32	0·73	8·63
1·0318	12·43	3·65	4·63	3·42	0·73	8·78
1·0322	12·18	3·40	4·65	3·41	0·72	8·78

TABLE III.

Normal Milk (Evening Meal).

Specific Gravity.	Total Solids.	Fat.	Sugar.	Proteids.	Ash.	Solids-not-Fat.
1·0320	12·61	3·65	4·85	3·37	0·74	8·96
1·0325	12·79	3·65	4·75	3·64	0·75	9·14
1·0335	12·39	3·55	4·87	3·28	0·69	8·84
1·0325	13·18	4·25	4·74	3·45	0·74	8·93
1·0327	12·35	3·55	4·72	3·35	0·73	8·80
1·0323	12·80	3·90	4·79	3·40	0·71	8·90
1·0320	12·83	4·00	4·85	3·27	0·71	8·83
1·0322	13·42	4·55	4·50	3·66	0·71*	8·87
1·0320	13·06	4·15	4·70	3·48	0·73	8·91
1·0307	12·44	4·00	4·48	3·26	0·70	8·44
1·0316	14·04	5·35	4·54	3·42	0·73	8·69
1·0322	12·25	3·55	4·66	3·29	0·75	8·70
1·0315	12·72	4·00	4·54	3·44	0·74	8·72
1·0313	12·20	3·55	4·54	3·38	0·73	8·65
1·0313	12·73	4·00	4·65	3·34	0·74	8·73
1·0323	12·98	4·10	4·78	3·34	0·76	8·88
1·0326	12·53	3·60	4·76	3·46	0·71	8·93
1·0322	12·53	3·75	4·66	3·38	0·74	8·78
1·0320	12·23	3·65	4·56	3·27	0·75	8·58
1·0325	12·39	3·50	4·96	3·22	0·71	8·89
1·0320	12·65	3·90	4·67	3·37	0·69*	8·75
1·0323	12·47	3·60	4·72	3·43	0·72	8·87

TABLE IV.

Abnormal Milk.

Specific Gravity.	Total Solids.	Fat.	Sugar.	Proteids.	Ash.	Solids-not-Fat.
1·0318	11·75	3·20	4·77	3·11	0·67*	8·55
1·0285	12·03	4·04	3·65	3·54	0·80	7·99
1·0298	12·53	4·14	3·89	3·68	0·82	8·39
1·0288	12·56	4·39	3·82	3·55	0·80	8·17
1·0295	12·23	3·94	3·84	3·63	0·82	8·29
1·0282	12·90	4·81	3·70	3·59	0·80	8·09

The first sample in the table of abnormal milks was obtained from cows the supply from which was stopped because they were suffering from vaccinia. The last five were yielded by the same herd at different times; the quantity in this case was very small.

The following figures were found in the last five samples:

Insoluble Ash.	Soluble Ash.	Chlorine in Ash.
0·45	0·35	0·160
0·47	0·35	0·160
0·45	0·35	0·155
0·46	0·36	0·162
0·45	0·35	0·160

For comparison I have given below (*a*) a sample of milk yielded by the cows, which gave the five abnormal samples, to which 15 per cent. of water had been added, and (*b*) a sample of normal milk containing 10 per cent. added water:

	Specific Gravity.	Total Solids.	Fat.	Sugar.	Proteids.	Ash.	Solids-not-Fat.	Insoluble Ash.	Soluble Ash.	Chlorine in Ash.
(*a*)	1·0243	10·63	3·74	3·06	3·13	0·70	6·89	0·42	0·28	0·139
(*b*)	1·0280	11·24	3·45	4·02	3·09	0·68	7·79	0·44	0·24	0·094

The average composition of normal milk deduced from the above analyses is:

	Specific Gravity.	Total Solids.	Fat.	Sugar.	Proteids.	Ash.	Solids-not-Fat.
Morning	1·0325	12·32	3·44	4·71	3·43	0·74	8·88
Evening	1·0322	12·71	3·90	4·69	3·39	0·73	8·81

The average composition of the five abnormal samples very low in solids-not-fat is:

1·0290	12·45	4·26	3·78	3·60	0·81	8·19

It is seen that though the solids-not-fat are very low, the proteids and ash are above the average, and the whole of the deficiency falls on the milk sugar. Comparison with the watered milks shows that the rule for discriminating between abnormal and adulterated milks has held with these samples. I do not think that any analyst would be misled into reporting these abnormal samples as watered milks.

I would point out that the above analyses of normal milks confirm Vieth's ratio of sugar, proteids, and ash of 13 : 9 : 2 to a remarkable degree. The ratio found is 12·8 : 9·3 : 2, which is in excellent agreement.

It will be remembered that two years ago Storch published a research on a new proteid in milk, and concluded that each fat globule was surrounded by a "mucoid"

* These ashes are probably too low, as the muffle in which they were incinerated was accidentally allowed to get red hot for a few minutes.

envelope; according to him, for each part of fat in a globule there were 0·38 parts of mucoid envelope, which was composed of mucoid proteid, ash, and water, but which contained no milk sugar. I then pointed out that, were this the case, cream should contain a smaller amount of milk sugar per 100 of water than the original milk, and this difference should be slightly more accentuated when the cream and separated milk are employed. I have already published figures to show that there is no evidence of the milk sugar in cream being low, and the following figures further bear out this view:

	Cream.	Skim Milk.	
	Per Cent.	Per Cent.	Calculated for 6·77 Per Cent. Solids-not-Fat.
Total solids	36·06	9·41	
Fat	29·29	0·12	
Sugar	3·47	4·81	3·49
Proteids	2·76	3·70	2·69
Ash	0·54	0·78	0·59
Solids-not-fat	6·77	9·29	6·77

It is seen that the sugar in the cream is almost identical with the amount calculated to be present in 6·77 per cent. solids-not-fat from the skim milk, and the difference in the proteids is very small. Were Storch's hypothesis correct, the quantity of milk sugar would be $\frac{63{\cdot}96 - 29{\cdot}29 \times 0{\cdot}38}{63{\cdot}96} \times 3{\cdot}49$, or 2·90 per cent.

As the evidence on which Storch concludes that a membrane exists round each fat globule—the behaviour of milk with ether and the staining of a layer—is evidence only of a layer of some kind, and not of a membrane; as Béchamp has, under rather different conditions, obtained results on treating milk with ether which are diametrically opposite to those of Storch; and as the analysis of cream gives no evidence of any decrease in the milk sugar, or marked increase in the proportion of proteids with the thickness of the cream, I am unable to see that there is any justification for the view that each globule in milk is surrounded by a mucoid membrane; certainly the question is far from being settled by Storch's results.

The average composition of clotted cream fround during 1898 was:

	Maximum.	Minimum.	Average.
Total solids	73·03	63·87	68·84
Fat	66·98	55·12	61·97
Ash	0·84	0·46	0·56
Solids-not-fat	10·29	5·51	6·87

In the table below are given the maximum, minimum, and average figures for percentage of water and refractive index of fat in butters:

Table V.

Composition of Butters.

Description.	Percentage of Water.			Refractive Index at 35°.		
	Maximum.	Minimum.	Average.	Maximum.	Minimum.	Average.
English fresh	14·41	11·96	13·21	47·2°	44·9°	46·0°
English salt	14·29	12·27	13·22			
French fresh	18·72	13·13	15·48	46·1°	44·6°	45·4°
Danish salt	15·62	9·97	13·32	46·0°	45·1°	45·6°

A butter prepared in the Channel Islands had a refractive index at 35° C. of 43·6°, which is the lowest I have yet met with; its Reichert figure was 15·8 c.c.

It is well known that when milk is passed through a separator, a white or brownish deposit is thrown on the sides of the drum. This is often talked of as "dirt," "putrid slime," etc.; as a matter of fact, though it contains the dirt in the milk, "separator-slime" is a product derived from milk.

The following are three analyses of this product:

Percentage obtained.	Total Solids.	Fat.	Milk Solids Calculated.	Other Solids-not-Fat.	Ash.	P_2O_5.
0·029	29·2	1·3	7·2	17·5	3·2	1·28
0·031	27·5	4·1	7·4	13·7	2·3	0·94
0·036	26·4	3·9	7·5	12·8	2·2	0·91

If the milk be clean this substance is obtained white in colour, and probably consists largely of Storch's mucoid. The high amount of phosphoric acid in the ash suggests the presence of a compound rich in phosphorus such as this, and the quantity of phosphoric acid found agrees fairly well with the view that practically the whole of the other solids-not-fat are mucoid.

Discussion.

The President (Mr. Fisher) said that he had been struck with the author's remark about the deficiency in non-fatty solids which had prevailed towards the latter part of the previous year. From his own experience, he was inclined to think that some deficiency had continued through the winter, and had not even yet disappeared in districts where the food was rather poor, and where the cows had not been liberally treated during the winter months.

Mr. Richmond said that, on the other hand, the non-fatty solids in the milk which had come to his laboratory during the last month or two had been quite normal.

THE ESTIMATION OF ALCOHOL AND ETHER IN THE PRESENCE OF PETROLEUM ETHER.

By H. Droop Richmond.

(*Read at the Meeting, April* 12, 1899.)

Some time ago I found that mixtures of ether and petroleum ether containing some alcohol were accumulating in the laboratory, and I wished to know the proportions of each, with a view to seeing the best mode of treating them.

The following methods were found satisfactory for the analysis of such mixtures.

Estimation of Alcohol.—It was found that the decrease of volume observed on shaking the mixture with water saturated with ether did not represent the alcohol, as petroleum ether dissolves ether from etherized water, and the figure obtained was a resultant of the quantity of alcohol removed from the mixture, and the quantity of ether added to it. By shaking the ethereal layer a second time with water saturated with ether, the petroleum ether dissolves a quantity of ether from the etherized water equal to that dissolved on the first shaking. The difference in volume of the ethereal layer obtained by the first and second separations gives the volume of ether dissolved

by the petroleum ether from the etherized water, and on subtracting this from the volume obtained by the first separation, the decrease of volume due to the alcohol being dissolved can be calculated.

The following procedure was adopted: Shake 20 c.c. of the mixture with 25 c.c. of water saturated with ether; note the volume of the ethereal layer (call this A). Run off the aqueous layer from the ethereal layer, and shake the latter again with 25 c.c. of water saturated with ether; note the volume of the ethereal layer after the second separation (call this B). Then $2A - B$ will give the volume of ether and petroleum ether, and $(20 + B - 2A) \times 5$ the percentage of alcohol by volume in the original mixture.

Estimation of Petroleum Ether.—Prepare a mixture of 20 c.c. sulphuric acid (90 per cent. H_2SO_4) and 20 c.c. glacial acetic acid, and cool. Place 10 c.c. of the mixture to be examined (or of the ethereal layer left after the estimation of alcohol) in a burette, add the mixture of acetic and sulphuric acids little by little, keeping the burette corked, and shaking well between each addition; the mixture should be cooled. When all is added, shake vigorously, and allow to separate. The layer separating is petroleum ether.

The following examples will show the accuracy of the method, and many other estimations as satisfactory have been made.

	Experiment I.	Experiment II.
Composition: Alcohol	9·1 per cent.	19·7 per cent.
Ether	45·5 ,,	27·1 ,,
Petroleum ether ...	45·5 ,,	53·2 ,,
Volume A	18·9 c.c.	17·2 c.c.
,, B	19·65 c.c.	18·3 c.c.
Alcohol deduced therefrom	9·25 per cent.	19·5 per cent.
Petroleum ether	44·5 ,,	53·0 ,,
Ether (by difference)	46·25 ,,	27·5 ,,

Petroleum ether can be recovered from these mixtures by adding sulphuric acid little by little and shaking well, the flask in which the experiment is performed being kept cool; the layer of petroleum ether which separates only requires washing and re-distilling for use. The acid liquid may be used for regenerating ether if it is thought worth while.

THE INFLUENCE OF AMMONIUM SALTS ON THE PRECIPITATION OF NICKEL BY AMMONIA.

By Arthur Marshall.

(*Read at the Meeting, June* 7, 1899.)

When ammonia in not too great a quantity is added to an aqueous solution of nickel ammonium sulphate, after a time a light green flocculent precipitate forms. When I first observed this, I thought the precipitate must be due to iron or other impurity in the nickel salt. On examination, however, I found that the precipitate consisted of a nickel compound. As this behaviour was not in accordance with my preconceived

ideas, I carried out a series of experiments to investigate the causes which affect the precipitation of nickel by ammonia.

Fresenius, in his "Qualitative Analysis," makes the following remark: "Ammonia added in small quantity to solutions of protoxide of nickel produces in them a trifling greenish turbidity; upon further addition of the reagent this re-dissolves readily to a blue fluid containing a compound of protoxide of nickel and ammonia." If the ammonia and the solution of the nickel salt be mixed in the cold, and be not allowed to stand long, the behaviour is in accordance with Fresenius's statement. But if the liquid be boiled, a very voluminous precipitate may be obtained under certain circumstances, even in the presence of considerable quantities of ammonium salts. This precipitate differs from that formed in the cold, in that it cannot be entirely re-dissolved even by a very large excess of ammonia. A very faint turbidity remains; so slight, however, that it can only be detected with difficulty.

Other text-books on qualitative analysis contain statements less correct than that of Fresenius. Valentin, Thorpe, and Clowes all state that the precipitate formed on adding a small quantity of ammonia to nickel solution has the composition $Ni(OH)_2$. On this subject I have collected the following evidence:

André (*Comptes Rendus*, 1888, cvi., p. 907), using nickel chloride and an insufficient quantity of ammonia, found the precipitate to have the composition $NiCl_2$, $8NiO$, $13H_2O$.

Habermann (*Monatshefte für Chemie*, 1884, v., p. 441), using nickel sulphate and dilute ammonia and boiling, obtained a precipitate which on analysis proved to be $NiSO_4.6NiO.10H_2O$. Similarly from nickel nitrate he obtained a precipitate of the composition $Ni(NO_3)_2.7NiO.5H_2O$.

I myself carried out the following experiment: Two grammes of nickel ammonium sulphate, $NiSO_4.(NH_4)_2SO_4.6H_2O$, were dissolved in 220 c.c. of water. To this was added 20 c.c. of a solution containing 17 grammes of ammonia per litre. The ammonia added was about twice that calculated as being necessary to convert all the nickel into $Ni(OH)_2$. The liquid was boiled for a short time, and the light green precipitate was filtered off, washed, and dried to constant weight at 110° C. It weighed about 0·35 gramme. On analysis it proved to have the composition $NiSO_4.8NiO.16H_2O$.

	Calculated.	Found.
Ni	5·66	5·74
SO_4	9·20	9·21
8NiO	57·53	57·65
$16H_2O$	27·61	
	100·00	

Calculation shows that nearly 60 per cent. of the nickel was precipitated.

I have performed some hundreds of experiments to observe the conditions which affect the precipitation of nickel by ammonia. I give below a few of them to indicate the results which may be expected. Solutions were prepared of nickel salts, of ammonium salts, of acids, and of ammonia, all either of normal strength, or some multiple thereof. These were measured out into a flask in various proportions, and mixed or boiled together and the result noted. *E.g.*, 5 c.c. of $\frac{N}{1}$ nickel chloride

solution (containing 29·5 grammes Ni per litre) were mixed with 50 c.c. of 4N (four times normal) ammonia (68 grammes NH_3 per litre). The mixture was then boiled, no precipitate was produced; 20 c.c. of distilled water were then added, and the liquid was again boiled, still no precipitate formed. Another 10 c.c. of water were added, on boiling a slight precipitate was produced. Ten c.c. of 4N ammonium chloride solution were then added and the solution boiled, but the precipitate was but little affected. The addition of another 10 c.c. had also little effect.

Five c.c. of $\frac{N}{1}$ nickel chloride were boiled with 5 c.c. of 4N ammonium chloride, and 4N ammonia was added gradually, the liquid being raised to boiling after each addition. When 1·5 c.c. had been added the solution was distinctly turbid. When 3·5 c.c. had been added there was a considerable quantity of precipitate and the liquid began to turn blue. The quantity of precipitate then decreased with further additions of the ammonia, until, when 16 c.c. had been added, there was hardly any left. The addition of 40 c.c. of distilled water had no perceptible effect on the liquid, but when 100 c.c. had been added it became very cloudy. On again adding ammonia the cloudiness diminished, but when 35 c.c. in all had been added there was still some precipitate in suspension.

Five c.c. of $\frac{N}{1}$ nickel chloride were mixed with 10 c.c. of 4N ammonium chloride and 5 c.c. of 4N ammonia. The liquid was kept boiling and water was added gradually. When 110 c.c. had been introduced the liquid became cloudy. The last experiment was repeated, using 5 c.c. of ammonium chloride instead of 10 c.c. The liquid turned cloudy when 75 c.c. of water had been added.

Five c.c. of $\frac{N}{1}$ nickel sulphate were mixed with 40 c.c. $\frac{N}{1}$ hydrochloric acid and 20 c.c. of 4N ammonia, and water was added as before. When 150 c.c. had been added there was a slight precipitate. Many more experiments might be given showing the effects of slight variations of the various conditions.

I have examined the relative influence of the following acids and their ammonium salts on the precipitation: hydrochloric, sulphuric, nitric, acetic, oxalic, and tartaric acids. It makes no difference whether the acid and ammonia be added separately or the equivalent amount of a solution of the salt be used. Nitric acid has about the same effect as the equivalent quantity of hydrochloric acid. Sulphuric and acetic acid have a slightly greater tendency to keep the nickel in solution. Tartaric acid has a considerably greater effect, and if added in sufficient quantity entirely destroys the violet colour of the solution, changing it to a pale green. The colour may as a rule be re-produced by the addition of more ammonia. In the presence of oxalic acid ammonia in dilute solution produces a slight cloudiness in the cold. This re-dissolves on heating, but after a short time a gray granular precipitate forms.

I have also performed a few experiments on solutions of salts of other metals. The three remaining elements of Group III. B, viz., zinc, manganese, and cobalt, behave very similarly to nickel. Zinc is not precipitated quite so readily as nickel, but there is no great difference. Cobalt requires a larger proportion of ammonium salt to keep it in solution, when small quantities of ammonia are added. It is also more readily precipitated by the addition of water. Manganese oxidizes so readily in alkaline solution that it is difficult to obtain results of even approximate accuracy.

Calcium, barium, and strontium do not of course give any precipitate with

ammonia. Magnesium gives a precipitate, but an equivalent quantity of ammonium salt or acid entirely prevents precipitation, or will even redissolve a precipitate already formed. Apparently a double salt, such as $MgSO_4.(NH_4)_2SO_4$, is formed in solution, and this gives no precipitate with ammonia.

Conclusions.—Nickel and the other metals of Group III. B give precipitates with ammonia, unless there is present a very large excess either of ammonia or of an ammonium salt or of both. These precipitates probably in all cases consist of basic salts containing several molecules of the oxide of the metal combined with one molecule of the salt and several molecules of water.

Ammonium salts have more influence than equivalent quantities of free ammonia in keeping the nickel in solution. The precipitation takes place more readily and completely in dilute than in concentrated solutions. Precipitation may in many cases be brought about by simply diluting with water and boiling.

I cannot lay down any general rule whereby one may be certain not to get any precipitation of these metals under any circumstances, for the presence in solution of other substances such as iron or alkali salts makes them more liable to precipitate. It may, however, be stated that 12 equivalents of ammonium salt and 12 of free ammonia should be present for each equivalent of any metal of Group III. B. It is, however, advisable to have more than this amount present. The solutions should not be very dilute.

ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

FOODS AND DRUGS ANALYSIS.

Areometric Determination of Fat in Milk. H. Timpe. (*Chem. Zeit.*, 1899, xxiii., 436, 455.)—On a previous occasion (*Chem. Zeit.*, 1894, xviii., 392) it was stated that this process was of wide application, but always gave results uniformly lower than those of the unmodified Soxhlet method, the difference being apparently proportional to the amount of fat at hand. To discover the reason for this peculiarity, the mechanism of the process has been carefully studied by noting the behaviour of filtered butter fat with the water and ether in the presence and also in the absence of the potassium hydroxide. From a large number of experiments on butters from different sources, it appears that a certain proportion of the material becomes saponified during the operation, and so does not pass into the ether. This proportion varies with the breed of the cows, with the amount and concentration of the alkali, and with the quantity of fat present; it is also dependent upon the temperature, wherefore the specified degree, 17·5° C., must not be departed from. The saponification represents no quantitative separation of the butter into its constituents; the olein is chiefly attacked, but the glycerides of solid fatty acids are also hydrolysed, and a repetition of the whole process produces further decomposition. For practical purposes it is necessary to accept some average value for this "saponification factor,"

and from four series of tests with Harz, Swiss, shop, and creamery butters, which gave 11·12, 9·89, 8·34, and 8·88 per cent. of saponified matter respectively, the author deduces a mean factor of 9·5 per cent.

It has been ascertained that no alteration of volume takes place when butter-fat is dissolved in ether, that the average specific gravity of the fat is 0·9520, and that the specific gravity of ether saturated with moisture, whether it be derived from pure water, or from dilute solutions of alkali metal hydroxides or carbonates, is constantly 0·7222 at 17·5° C. The usual figures given for the mutual solubility of ether and water do not prove quite correct, therefore special experiments have been made to determine the volumes produced by agitating known quantities of wet ether and water together. Collating the results, the formulæ for calculating the amount of fat in milk are somewhat modified from those given in 1894, and now become:

$$(1)\quad Z = A - 0{\cdot}075W.$$

$$(2)\quad F = 1{\cdot}4566Z\frac{S - 0{\cdot}7222}{(0{\cdot}9520 - S)\frac{vs}{100}},$$

where A is the weight of water-saturated ether taken, W the weight of water, Z the weight of aqueous ether left after agitation, S the specific gravity of the ethereal solution of fat, v the volume and s the specific gravity of the original milk, and F the weight of the fat it contains. The figures deduced from these formulæ for the quantity of fat in milk show a maximum difference of 0·04 per cent. from the Soxhlet process, and among themselves the several tests of one sample give a maximum difference of 0·035 per cent. The formulæ also exhibit the advantage that a small sample of milk can be analysed, since the volume of ether can be reduced at will. The method is equally available with milks containing more than 5·5 per cent. of fat. F. H. L.

Microscopy of Butter. A. Zega. (*Chem. Zeit.*, 1899, xxiii., 312.)—In order to obtain rapidly an idea as to the genuineness of a specimen of butter, and in the absence of a refractometer, the following process is suggested: The sample is melted and filtered into a test-tube, which is kept for two minutes in a boiling water-bath. By means of a hot pipette 1 c.c. is measured into a 50 c.c. stoppered tube containing 20 c.c. of a mixture of 6 parts of ether, 4 of alcohol, and 1 of glacial acetic acid. The whole is well shaken, and allowed to cool in water at 15° or 18° C. Pure butter remains clear, and only gives a slight deposit after standing one or one and a half hours. Margarine shows a deposit in one or two minutes, and in ten minutes yields a copious precipitate. Mixtures of butter with 10 per cent. of margarine begin to separate in about fifteen minutes. As soon as a few solid particles have fallen, they are withdrawn and examined under the microscope. Genuine butter appears in long, very narrow crystalline rods, often pointed at the ends, sometimes bent, and usually joined together centrally into more or less symmetrical open stars. Margarine crystals consist of bundles of minute needles packed closely together into circles, sheaves, or dumb-bell-like masses.

Similar experiments with lard and tallow are not so satisfactory. Pure lard crystallizes as broad rods with almost square ends, often cohering into stars and

brushes; tallow looks exactly like margarine; but when the two fats are present, the above process is not trustworthy. The best results are obtained by preparing the crystalline deposit as before, holding the tube for five minutes in water at 36° or 40° to effect partial re-solution, then permitting a second crystallization to take place at the ordinary laboratory temperature. Pure lard crystals thus appear as already described; 5 per cent. of tallow is shown by the presence of the sheaves or dumb-bells, while the lard plates are caused to look much smaller. F. H. L.

Detection of Caramel in Spirits and Vinegar. C. A. Crampton and F. D. Simons. (*Jour. Amer. Chem. Soc.*, xxi., 355-358.)—The authors make use of the well-known colour-absorbing power of Fuller's earth to distinguish between the natural colour which spirits acquire on storing in wood, and the artificial colour imparted by the addition of caramel. The earth appears to have a stronger affinity for the artificial than for the natural colouring matter. In the experiments cited, the difference in colour before and after treatment was determined by means of Lovibond's tintometer. Forty samples of spirits, which were known to be naturally coloured, furnished the following results:

	Per Cent. of Colour removed.
Maximum	25·0
Minimum	8·3
Average...	14·6

Whereas eighteen samples known to be artificially coloured gave the following figures:

	Per Cent. of Colour removed.
Maximum	54·1
Minimum	40·0
Average...	44·7

A sample of water-white spirits was coloured with caramel and treated with Fuller's earth, with the result that 70 per cent. of the colour was removed. It is necessary that the tests should always be carried out under precisely similar conditions and with the same earth, as the colour-absorbing power of different samples varies considerably. The test also appears to be applicable to vinegar.

H. H. B. S.

Presence of Barium Salts in Cayenne Pepper. H. Kaiser. (*Chem. Zeit.*, 1899, xxiii., 496.)—In the course of a discussion which has been proceeding lately in the *Chem. Zeit.*, as to whether an abnormal proportion of barium in a vegetable powder may be explained by the living plant having absorbed barium salts from the soil in which it was grown, the author remarks that this is impossible, inasmuch as barium in sufficient quantity is a vegetable poison. Cayenne occasionally contains the metal simply because, in order to give the substance a brilliant fiery colour, it is adulterated with a barium-ponceau lake; and the barium exists as sulphate or carbonate according to the composition of the base on which the colour is stained.

F. H. L.

The Assay of Belladonna Leaves and Some of its Preparations. F. X. Moerk. (*Amer. Journ. Pharm.*, 1899, lxxi., 105-120.)—After numerous experiments the author has found the following modification of Keller's process to effect a complete extraction of the alkaloids from a drug or preparation, although it is not a quick method.

The moisture is determined in 2 grammes of the powdered leaves at 100° C., without previous drying over lime or sulphuric acid.

To determine the extract 20 grammes of the powdered leaves are introduced into a small percolator (previously plugged with cotton wool) together with 50 c.c. of a mixture of 2 volumes of alcohol and 1 volume of water, and the mass stirred with an iron wire until the mixture is homogeneous. The sides of the percolator and the wire are then washed with 25 c.c. of the solvent and the whole allowed to stand for five or six hours. Percolation is then allowed to proceed until 75 c.c. have passed through, when the maceration is renewed for five or six hours. The alternate maceration and percolation are continued until 300 c.c. of the solvent have been used. By evaporating the percolated liquid in a weighed dish until the residue can no longer be stirred with a glass rod the yield of extract is obtained.

The extract thus obtained (or about 5 grammes of ordinary belladonna extract) is stirred with 10 c.c. of acidulated water (2 c.c. of U. S. P. sulphuric acid to 500 c.c. of water), the disintegrated mass transferred to a separating funnel, and the dish washed with small portions (2 c.c.) of acidulated water. The insoluble residue of chlorophyll, etc., is also washed in by means of three successive portions of 10 c.c. of a mixture of ether and chloroform (4 : 1 by weight). The dish is again washed out with the acidulated water until 25 c.c. in all of this has been used, the washings in each case being added to the liquid in the separator. Finally 20 c.c. more of the mixture of chloroform and ether are added, the whole shaken thoroughly, and after separation of the liquids the acid solution run off.

The shaking out is repeated with 15, 10, and 5 c.c. of the acidulated water, and the united acid solutions shaken in a separating funnel with 25 c.c. of a mixture of 2 parts of ether and 4 parts of chloroform by weight, and 8 c.c. of ammonium hydroxide (10 per cent.). The ether-chloroform layer is transferred to a smaller separating funnel and the extraction of the aqueous residue repeated with 15, 10, and 5 c.c. successively of the same solvent.

The author found that emulsification of the alkaloidal solution at this stage could readily be prevented by adding a fragment of stearic acid (15 to 20 milligrammes) little by little.

The ether-chloroform extract is filtered from the small separating funnel into a dry flask, and the solvent evaporated on the water bath until all odour of chloroform disappears. The residue is dissolved in 5 c.c. of ether, which is evaporated, and the residue again dissolved in 5 c.c. of ether, which is again evaporated. Finally, the residue is dissolved in 8 c.c. of neutral alcohol, 30 c.c. of water added, and the alkaloids titrated with standard hydrochloric acid, with hæmatoxylin solution as indicator.

The results obtained by this process with different commercial varieties of belladonna leaves were:

	Leaves.			Extract.
	Moisture Per Cent.	Extract Per Cent.	Alkaloids Per Cent.	Alkaloids Per Cent.
English, cultivated (Allen's)	8·40	*a.* 33·55 *b.* 32·55	*a.* 0·5996 *b.* 0·6054	*a.* 1·8510 *b.* 1·8280
German, cultivated ...	7·60	*a.* 36·55 *b.* 36·30	*a.* 0·5155 *b.* 0·5050	*a.* 1·5980 *b.* 1·5850
German, wild	6·80	*a.* 27·00 *b.* 27·45	*a.* 0·4997 *b.* 0·4997	*a.* 1·6998 *b.* 1·7133

The author points out that the yield of extract obtained from the leaves is from 20 to 24 per cent. higher than the yield usually published, and attributes this to the larger amount of solvent used in the extraction. C. A. M.

The Analysis of Commercial Veratrine. G. B. Frankforter and L. B. Pease. (*Amer. Journ. Pharm.*, 1899, lxxi., 130-133.)—The commercial products sold under the name of "veratrine" show a great variation, which is to be attributed partly to the difficulty with which the alkaloids, obtained from various species of *veratrum*, crystallize, and partly to the fact that several of these alkaloids have been known by the name of "veratrine."

Thus, Couerbe gave the name to the most important of the three substances which he isolated—an alkaloid with the formula $C_{34}H_{43}N_2O_6$. Merck assigned to veratrine the formula $C_{32}H_{52}N_2O_8$, and Wiegelin the formula $C_{52}H_{86}N_2O_5$. Wright and Luff (*Journ. Chem. Soc.*, xxxiii., 338) have isolated three bases, to one of which ($C_{37}H_{49}NO_9$) they assign the name of "veratrine," although the most abundant of the three is cevadine ($C_{32}H_{49}NO_9$). Hence three separate alkaloids have been known as "veratrine," any one of which might have been taken as official.

The authors have examined a number of specimens of commercial veratrine, and have found cevadine to be the predominating base in each case. In no instance was more than a trace of either of the other substances known as "veratrine" discovered.

The melting-points of the different samples examined varied slightly, although there was a comparative agreement in the general physical and chemical properties. On treating the drug with ether, the greater part dissolved, forming a colourless solution. The small quantity of insoluble matter was an amorphous powder, melting at 184° to 285° C. It did not fully answer the description of either of the other bases.

The following table gives the percentage of insoluble substance, and its melting-point in the case of the veratrine recently prepared by different manufacturers:

	Merck.	Tromsdorff.	Powers and Weightman.	Mahlenkrodt.
Insoluble substance, per cent. ...	0·86	3·89	3·6	3·26
Melting-point, ° C.	184°	285°	206°	231°

The soluble portion was identical with Wright's cevadine, as is seen by a comparison of the results of the analyses:

Calculated for $C_{32}H_{49}NO_9$.	Merck. No. 1.	Merck. No. 2.	Tromsdorff.	Powers and Weightmann.	Mahlenkrodt.
C = 64·96	64·6	64·5	64·72	64·74	64·82
H = 8·29	8·4	8·6	8·36	8·19	8·41
Melting-point	148° C.		147° C.	148° C.	148° C.

Two samples of the drug prepared some years ago were found to consist of a mixture of several substances, resembling the "veratria" prepared by Pelletier and Caventou in 1819 (*Ann. Chim. Phys.* (II.), xiv., 69). C. A. M.

TOXICOLOGICAL ANALYSIS.

Microscopic Identification of Blood. C. Strzyzowski. (*Oesterr. Chem. Zeit.*, 1899, ii., 334.)—A reagent, consisting of glacial acetic acid, mixed with 1·5 or 2 per cent. by volume of hydriodic acid, is prepared freshly each time it is wanted. The suspected sample is laid on a microscope slide, covered with a very small cover-glass, and a drop of the acid is applied to the edge in order that it may penetrate between the glasses. The liquid is boiled off over a minute spirit flame, and the residue is examined optically. From new or dried blood, the hæmatin crystals appear large and well formed in the shape of rhomboidal plates, with their characteristic coal-black colour. The process will detect 0·025 milligramme of fresh blood.

In those rare cases where the above acid mixture cannot be employed, a glycerin-sulphuric acid reagent may be substituted. This is prepared from 10 c.c. of glycerin and 2 or 3 drops of strong sulphuric acid. A cover-glass is moistened therewith, laid on the object as it rests on a slide, the liquid boiled for eight or twelve seconds, and examined with a power exceeding 400 diameters—preferably an immersion lens.

F. H. L.

The Detection and Estimation of Bromoform in Toxicological Work. A. Richaud. (*Journ. Pharm. Chim.*, 1899, ix., 232-236.)—The method commonly used for the detection of chloroform consists in decomposing it at a red heat into chlorine and hydrochloric acid, and collecting these in a solution of silver nitrate. In the case of bromoform, however, this method is unsatisfactory except as a qualitative test when large quantities are present. When the amount of bromoform does not exceed 50 centigrammes, the author finds that not more than 60 or 70 per cent. of the theoretical quantity can be recovered in the form of silver nitrate.

It is possible to completely remove bromoform from organic matter by means of a current of steam, the whole of it passing over with the first portions of the distillate, the total quantity of which need not exceed 75 to 100 c.c.

In order to estimate the bromoform thus recovered, the author has made experiments with various methods. Greshoff has described a process of estimating iodoform based on its decomposition with silver nitrate at a low temperature,

$$CHI_3 + 3AgNO_3 + H_2O = 3AgI + 3HNO_3 + CO,$$

but the author was unable to bring about a corresponding reaction with bromoform even by boiling the liquid under a reflux condenser.

By gently heating bromoform with alcoholic potassium hydroxide it is decomposed in accordance with the equation

$$CHBr_3 + 4KOH = 3KBr + KHCO_2 + 2H_2O.$$

The reaction is rapid, and when complete the alcohol is removed by evaporation, the residue neutralized with acetic acid, and the bromide determined volumetrically, or even gravimetrically if the solution be well cooled in order to prevent the reducing action of the formate. The author has obtained almost theoretical results by either method.

Like chloroform, bromoform is decomposed in the cold by an aqueous solution of potassium hydroxide, with the formation of potassium bromide, carbon monoxide, and water:

$$CHBr_3 + 3KOH = 3KBr + CO + 2H_2O.$$

The reaction takes place less readily with bromoform than with chloroform, but only a slight elevation of temperature is required to complete it in a relatively short time. The mixture of bromoform and aqueous potassium hydroxide is gently heated by means of a low spirit flame under a reflux condenser for about an hour and a half, and the resulting bromide determined in the usual manner. The author regards this as the most satisfactory method of estimation.

When bromoform is to be detected or estimated in substances which contain certain products of putrefaction or in fæcal matter, it is necessary to take into account the volatile substances which are simultaneously removed by the steam, and which reduce the silver nitrate added to precipitate the bromide. In such cases correct results may be obtained by evaporating the liquid containing the potassium bromide and impurities to dryness, and slightly igniting the residue before making the estimation. C. A. M.

ORGANIC ANALYSIS.

The Detection of Methyl Alcohol in Ethyl Alcohol. A. Trillat. (*Ann. de Chim. Anal.*, 1899, iv., 42-44.)—The author has simplified and rendered more sensitive the method described by him in a former paper (ANALYST, xxiv., 13). In the modified process a quantity of the alcohol to be tested, corresponding to 10 c.c. of absolute alcohol, is diluted to 150 c.c., and mixed with 70 c.c. of sulphuric acid (1 : 5) and 15 grammes of powdered potassium bichromate. After being shaken at intervals for twenty minutes the liquid is distilled, the first 25 c.c. being rejected, and the distillation continued until 100 c.c. have been collected.

Fifty c.c. of this distillate are mixed with 1 c.c. of rectified dimethyl-aniline, and transferred to a 75 c.c. flask, which is carefully closed with a ground-stopper and placed in a water-bath kept at 70° to 80° C. An occasional shake is given, and after three hours the condensation is complete.

The liquid is then rendered distinctly alkaline with sodium hydroxide, and the dimethyl-aniline distilled, the distillation being stopped when 25 c.c. have passed over.

The residue in the flask is acidified with acetic acid and shaken, and a few c.c. tested for methyl alcohol by adding 4 or 5 drops of water with lead dioxide in suspension (about 1 gramme in 100 c.c.) and boiling. If it be present a blue coloration

is formed during the oxidation, which may be compared with the colours yielded under the same conditions by standard typical solutions of methyl alcohol.

By this modification the author states that it is possible to detect as little as 0·2 per cent. of methyl alcohol in ethyl alcohol, whereas with the original method the limit of sensibility was about 1 per cent. By a preliminary fractionation of the liquid he has readily detected 1 part in 1,000.

The only precautions to be observed are to have the dimethyl-aniline quite pure, and to remove it completely from the liquid after the condensation.

C. A. M.

On the Detection of Methyl Alcohol in Commercial Spirits. A. Trillat. (*Journ. Pharm. Chim.*, 1899, ix., 372-375.)—As many of the spirits of commerce contain esters, colouring matters, and other substances which interfere with the application of the author's method of detecting methyl alcohol (see preceding abstract), it is necessary to treat the sample in the following manner before applying the test. Fifty c.c. of the liquid are mixed with 50 c.c. of water and 8 grammes of lime, and fractionally distilled with the aid of a Glinsky bulb-tube. The first 15 c.c. of the distillate are diluted to 150 c.c., mixed with 15 grammes of potassium bichromate and 70 c.c. of sulphuric acid (1 : 5) and left for an hour. The condensation with dimethyl-aniline and the oxidation are then carried out as described before.

In the case of absinthe, and, in general, of liqueurs which give a precipitate on dilution with water, 50 c.c. are mixed with an equal volume of water and 2 to 3 grammes of slaked lime, filtered through purified animal charcoal, and the colourless filtrate distilled.

The author has tested a large number of authentic samples of different kinds of spirit without finding any trace of methyl alcohol, but in several commercial samples, notably absinthe, kirsch, and rum, from 5 to 15 per cent. was present, which had evidently been fraudulently added.

Samples of Jamaica and Martinique rum and of cognac were compared with standard samples containing $\frac{1}{500}$ of methyl alcohol without giving any sign of the blue coloration. These results confirm those of P. Geerligs (*Bull. de l'Ass. des Chim. de Sucrerie*, 1898, No. 11).

On the other hand, certain genuine samples of brandy (*Marcs*) were found to contain about 0·25 per cent. of methyl alcohol, but as this was not an invariable constituent, the author suggests that its presence in these instances might have been due to defective distillation.

C. A. M.

A Simple Colour Reaction for Methyl Alcohol S. P. Mulliken and H. Scudder. (*Amer. Chem. Jour.*, 1899, xxi., 266.)—The test proposed depends upon the oxidation of methyl alcohol to formic aldehyde and the detection of the latter by its reaction with resorcinol and sulphuric acid. It is carried out as follows: A spiral copper wire is heated in the upper part of the flame of a Bunsen burner, and, while at a bright red heat, is plunged into 3 c.c. of the solution to be examined. If the solution be very dilute, this procedure must be repeated three or four times, and

in most cases it is desirable to repeat it at least once. Concentrated spirit should always be diluted before oxidation with the hot spiral. One drop of a 0·5 per cent. aqueous solution of resorcin is then added, and the mixture poured cautiously into a test-tube containing a few c.c. of concentrated sulphuric acid. Methyl alcohol is indicated by the production of a rose-red zone at the junction of the two liquids; but in and above this zone a scanty white or pinkish coagulum appears, which, on standing, increases in colour, and finally separates and rises in purplish-red flocks. The use of too large a quantity of resorcinol tends to destroy the purity of the rose colour, thus rendering the test less delicate. It is pointed out that the more common methyl esters and ethers are all slightly hydrolysed when dissolved in water, and consequently, as such solutions contain free methyl alcohol, they give this reaction.

The only other compounds which were found to give anything approaching to the reaction were the secondary and tertiary butyl alcohols, dimethyl-ethyl-carbinol, and formic acid. In the case of the secondary and tertiary alcohols, however, the pink zone is separated from the acid by a zone of lemon-yellow, and the red colouring matter does not separate in flakes. The coloration produced by formic acid is very faint; but to guard against mistakes arising from this cause, it is recommended that mixtures which react strongly acid should be neutralized with sodium carbonate before distillation. (*Cf.* ANALYST, xxii. 96.) H. H. B. S.

The Volumetric Estimation of Acetic Aldehyde. X. Rocques. (*Ann. de Chim. anal.*, 1899, iv., 13-17.)—The proposed method consists in treating the liquid containing the aldehyde with an excess of sulphurous acid or a bisulphite and titrating the reagent left uncombined by means of a standard solution of iodine.

For the process finally adopted as satisfactory the reagents required are: (1) A solution prepared by dissolving 12·6 grammes of dry sodium sulphite in 400 c.c. of water, adding 100 c.c. of sulphuric acid, and sufficient 96 per cent. alcohol to make a litre, and filtering from any crystals of sodium sulphate which may deposit. (2) A decinormal solution of iodine in potassium iodide. One c.c. of this corresponds to 0·0032 gramme of sulphurous acid and 0·0022 gramme of acetic aldehyde. The first solution is titrated with the second, and if the sulphite used was pure, 10 c.c. of the former should require 20 c.c. of the latter.

In the estimation of the aldehyde, 10 c.c. of the liquid under examination are placed in a long-necked flask with a mark at 100 c.c. Fifty c.c. of the sulphurous acid solution are added, the liquid made up to the mark with pure 50 per cent. alcohol, the whole well shaken, and the flask securely corked.

A blank determination is made at the same time by placing 50 c.c. of the sulphurous acid solution in a similar flask and diluting it with alcohol to 100 c.c.

The two flasks are immersed in a water-bath kept at 50° C. for four hours, after which their contents are cooled, 50 c.c. taken from each and titrated with the standard iodine solution, and the difference calculated into the corresponding quantity of acetic aldehyde.

If the liquid to be tested contain less than 1 per cent. of aldehyde, the reagents must be diluted. If the proportion of aldehyde be 0·5 per cent., the sulphurous acid

solution should be diluted with its own volume of 50 per cent. alcohol and $\frac{N}{10}$ iodine solution used; whilst with a percentage of 0·1 both solutions should be diluted to one-tenth their strength.

As sulphurous acid does not combine with paraldehyde the presence of that substance in the liquid does not affect the result, though obviously other aldehydes must not be present.

The following results are given to show the accuracy of the method:

Ethyl aldehyde in 10 c.c. of alcohol—

	Grammes.						
Taken	... 0·312	0·279	0·100	0·100	0·075	0·050	0·025
Found	... 0·310	0·277	0·094	0·093	0·074	0·048	0·023

C. A. M.

The Halphen Reaction for Cotton-seed Oil. D. Holde and R. Pelgry. (*Chem. Rev. Fett. u. Harz. Ind.*, vol. vi., pp. 67, 68; 94, 95.)—The authors found that this reaction ceases to occur when the oil has been previously heated to 250° C. for a short time, and on the publication of Soltsien's results (*Zeits. öffentl. Chem.*, 1899, p. 106), according to which a temperature of 200° C. does not affect the susceptibility of the oil towards Halphen's reagent, they repeated their experiments at 210° and 250° C., with the result that the limit of sensitivity of the oil appears to lie between these temperatures. They point out that, although the probability is remote that heated cotton-seed oil would be used for adulterating edible oils, on account of its disagreeable flavour, this objection does not apply to oils for technical purposes.

C. S.

The Composition and Analysis of Acetone Oil. A. and P. Buisine. (*Journ. Pharm. Chim.*, 1899, ix., 375-377.)—The authors make use of the following method for the examination of acetone oil as manufactured from the washings of the *desuintage* of wool.

The density is determined with a hydrometer at 15° C. In the case of suint acetone oils it varies from 0·830 to 0·835.

For the determination of the solubility in water 10 c.c. of the oil are shaken for a few minutes in a stoppered graduated tube, with 40 c.c. of water, the tube allowed to stand, and the layer of acetone oil which separates read off. Suint acetone oil dissolves to the extent of from 77 to 82 per cent., the soluble portion being composed almost exclusively of primary ketones—dimethylketone and methylethylketone.

Its solubility in sodium bisulphite solution (30 Bé.) is determined by shaking 100 c.c. of the sample with 350 c.c. of the bisulphite solution at intervals during several hours, allowing the layers to separate, decanting the insoluble portion and measuring its volume. As the insoluble portion only amounts to from 6 to 9 per cent. it follows that suint acetone oil consists almost entirely of compounds of a ketonic nature, capable of combining with sodium bisulphite.

In the distillation of the oil the same quantity (250 c.c.) is always distilled under the same conditions and special note taken of the quantity distilling between 70° and

90° C. This fraction in suint acetone oil amounts to from 74 to 80 per cent., consisting for the most part of ethylmethylketone. The proportion of dimethylketone is very small, and never exceeds 5 per cent.

The results thus obtained with three samples of suint acetone oil of different origin were:

	I.		II.		III.	
Density at 15°	0·833		0·835		0·835	
	Per Cent.		Per Cent.		Per Cent.	
Portion insoluble in water	20		21·5		18	
Portion insoluble in sodium bisulphite	8		9		6·5	
Distillation, from 60° to 70° C. ...	4		1		4	
,, ,, 70° to 76° ...	52	from 70° to 90°, 78 per cent.	50	78 per cent.	51	76 per cent.
,, ,, 76° to 80° ...	10		16		11	
,, ,, 80° to 85° ...	10		8		7	
,, ,, 85° to 90° ...	6		4		7	
,, ,, 90° to 100° ...	7		8		10	
,, ,, 100° to 110° ...	4		5		6	
,, above 110° ...	7		8		4	
	100		100		100	

The heat of combustion determined in Mahler's bomb varied from 7,850 to 7,900 calories for 1 gramme of crude oil.

The authors state that they have found a great regularity in the nature of the products obtained in the manufacture as well as in the amount of the yield.

A sample of acetone oil was prepared from crude calcium acetate (pyrolignite), the yield being scarcely 5 per cent. of acetone. On analysis this crude product gave the following results:

Density at 15° C.	0·842	
	Per Cent.	
Portion insoluble in water	44	
Portion insoluble in sodium bisulphite	9	
Distillation from 60° to 70° C. ...	0	From 70° to 90°, 36 per cent.
70° to 76°	2	
76° to 80°	10	
80° to 85°	12	
85° to 90°	12	
90° to 100°	28	
100° to 110°	10	
above 110°	26	
	100	

The pyroligneous acetone oils differ greatly in composition from the suint oils. They are much poorer in ketones, distilling below 90° C., especially ethylmethylketone, which is only present to about half the extent; but, on the other hand, they contain a far greater proportion of higher ketones, distilling above 100° C.

C. A. M.

Notes on Glycero-phosphoric Acid. J. Cavalier and Pouget. (*Bull. Soc. Chim.*, 1899, xxi., 364-366.)—Since Pelouze has shown that the glycero-phosphates of metals of the alkaline earths are less soluble in hot than in cold water, and are precipitated from a saturated solution on boiling, the authors have made experiments to determine the solubility of calcium glycero-phosphate in water at different temperatures. Their results show that 100 grammes of the solution contain the following quantities of anhydrous salt at the temperatures named:

C°.	Calcium Glycero-phosphate. Grammes.
16	7·9
36	4·4
51	2·3
77	1·3
86	1·25
100	1·15

The precipitate deposited on heating the solution is crystalline when it is formed slowly from a not too concentrated solution. It can be readily filtered and washed, and is completely soluble in water. The stability of the salt in solution resembles that of ethyl and methyl-phosphates. The authors prefer to purify calcium glycero-phosphate by precipitating it by heating its aqueous solution to 60° or 70° C. rather than by the addition of alcohol. They consider that little is gained by boiling the liquid.

Barium glycero-phosphate behaves differently, and its solutions are readily decomposed by heat, hence the method of purification by boiling the liquid is not suitable for the barium salt, and it is preferable to separate it by means of alcohol.

The method described by Cavalier (*Compt. Rend.*, cxxvii., 60) for the volumetric determination of esters of phosphoric acid in the presence of free phosphoric acid is also applicable to the determination of mixtures of glycero-phosphoric acid and free phosphoric acid. A solution of barium hydroxide is run into the cold solution until the colour of methyl-orange is changed. The liquid is then heated, and the titration completed with phenolphthalëin as indicator. The insoluble barium phosphate is filtered off, and the hot filtrate should be sufficiently dilute to retain the barium glycero-phosphate in solution. C. A. M.

The Composition and Reactions of Solanine. P. Cazeneuve and P. Breteau. (*Journ. Pharm. Chim.*, 1899, ix., 465-468.)—There is a considerable difference in the formulæ and melting-points assigned by different chemists to solanine. According to Zwenger and Kind it melts at 240° C., and has a composition corresponding with the formula $C_{43}H_{71}NO_6$. Kletzinsky gives the same melting-point, but the formula $C_{21}H_{35}NO_7$; Hilger states that it melts at 235° C., and has the formula $C_{42}H_{67}NO_{15}$; and according to Firbas its formula is $C_{52}H_{93}NO_{18}$, and its melting-point 244° C.

In the authors' opinion these differences are due to the methods of extraction adopted, which they consider are likely to bring about profound alterations in the solanine.

They recommend the following method of extracting the glucoside as the most suitable for obtaining it without alteration. The potatoes are allowed to germinate

protected from sunlight, and the shoots not exceeding 10 centimetres in length are intimately ground up with half their weight of slaked lime.

The mass is dried in the air at the ordinary temperature and extracted with cold 93 per cent. alcohol. The alcoholic extract, which has a slightly yellow colour, is evaporated *in vacuo* at 40° to 45° C. to the consistence of a syrup. On cooling this forms a crystalline mass, which is washed with petroleum spirit and ether, and crystallized three times from boiling 95 per cent. alcohol.

By this process the authors obtained a yield of about 0·5 gramme of solanine from a kilogramme of germs. It formed absolutely colourless needle-shaped crystals, which were insoluble in water and ether, soluble with difficulty in cold alcohol, but more easily soluble in boiling alcohol. It had a very bitter taste and a slightly alkaline reaction. It melted at 250° C., and had an elementary composition agreeing with the formula $C_{28}H_{47}NO_{10}.2H_2O$.

When hydrolysed by means of hydrochloric acid it yielded a crystalline product which dissolved in ether and melted at 190° C. (characteristics of solanidine), and a reducing sugar which furnished an osazone.

This solanine also differed from the products hitherto described in the nature of its colour reactions with nitric, hydrochloric, and sulphuric acids. With sulphuric acid (monohydrate) it was coloured but slightly yellow, the colour being chiefly at the edges and changing to faint rose and subsequently to violet. The solanines hitherto prepared give, on the contrary, pronounced colorations passing from orange to dark violet and brown. With nitric acid (specific gravity 1·5) the authors' solanine formed a colourless solution, which only became faint rose after a very long time, whereas in the case of other solanines the colourless solution rapidly assumes a purple coloration which soon disappears.

Hydrochloric acid (specific gravity 1·171) is usually stated to give a yellow colour with solanine, but the authors' glucoside remained absolutely colourless. On adding to their product drop by drop a mixture of sulphuric acid (6 parts) and absolute alcohol (9 parts) while still warm, the authors found that the crystals became bright green while the liquid surrounding them became a very faint rose colour. In the case of a solanine of German origin this reagent gave a blood-red coloration. C. A. M.

Determination of Unaltered Cellulose in Gun-cotton. G. Lunge and E. Weintraub. (*Zeits. angew. Chem.*, 1899, 473.)—The following process forms the concluding portion of a lengthy article upon the general characteristics of nitro-cellulose. It is based on the fact that an alcoholic solution of sodium ethylate decomposes the nitro-compounds, leaving the ordinary cotton intact. Two or 3 grammes of metallic sodium are dissolved in 100 c.c. of alcohol, the solution is filtered if necessary, and 100 c.c. of acetone are added; 150 c.c. of the reagent are mixed with 5 grammes of the cotton in a flask or basin at a temperature of 40° or 50° C., and stirred or shaken for twenty or thirty minutes. The mass is allowed to settle, the liquid poured off through a small filter, and the residue rinsed in alcohol; the precipitate is treated with water till the brown substance dissolves, the insoluble matter is brought on to the paper, and washed with hot water and very weak hydrochloric acid. If the

residual non-nitrated cellulose is large in amount, and if extreme accuracy is not required, this first product may be considered pure; for it will evolve no gas in the nitrometer, though giving a strong reaction with diphenylamine. To extract the last traces of nitro-cellulose, it should be treated again at the above temperature with 50 c.c. of the sodium ethylate and acetone reagent, and then filtered through a tared paper dried at 100° C. The product is almost colourless, but may be completely bleached with weak acid calcium hypochlorite. The process can be simplified by allowing the first action to take place in the cold for fifteen minutes to three or four hours according to the proportion of ordinary cotton expected, heating the second time as described.

The above scheme is applicable both to explosive cotton and to collodion cotton: substitution of the acetone by a mixture of ether and alcohol renders the analysis more troublesome and less accurate. A rough idea of the quantity of cellulose in explosive cotton may be obtained by merely dissolving it in acetone: more than 0·85 per cent. of unaltered cotton is indicated by insoluble fibres floating upon the top of the liquid. F. H. L.

Valuation of Indigo. W. Holdschmidt. (*Zeits. angew. Chem.*, 1899, 451, 475.)—The usual process for valuing indigo by heating it on the water-bath with strong sulphuric acid and then titrating the sulphonic acid with permanganate, presents two difficulties: (*a*) If the temperature rise too high, carbonaceous matter and sulphurous acid may be formed; and (*b*) the end point of the reaction is very troublesome to determine by simply watching the colour of the liquid, especially in such grades of indigo as contain much indigo red. These difficulties can be overcome by sulphonating in the cold with concentrated sulphuric acid mixed with 40 per cent. of phosphoric anhydride: 0·5 to 1 gramme of the finely powdered dry indigo is weighed into a tube, covered with 10 c.c. of the acid mixture, stirred at intervals with a glass rod, and kept in the desiccator for two hours or over-night. The mass is then poured through a funnel into a 500 or 1,000 c.c. flask filled one-third with water, the tube well rinsed out with cold water, the solution made up to the mark, and filtered quickly through a double paper. Twenty-five c.c. of the filtrate are next diluted with 100 or 125 c.c. of water, and titrated in a porcelain basin with centi-normal permanganate which has been standardized on oxalic acid of equivalent strength. The reagent is added at the rate of 120 or 130 drops per minute till the liquid is only faintly green, and the permanganate produces a dark cloud where it mingles with the indigo solution. Four more drops are run in with very gentle agitation of the liquid; the cloud is watched for, and when it completely disappears, the volume of permanganate is read off and 0·2 c.c. (corresponding to the 4 drops) deducted from the total. The amount of water, the speed of adding the reagent, and the illumination of the flask must be kept uniform in all tests. It is convenient to work by means of a powerful paraffin lamp.

The process employed by Donath and Strasser (*Zeits. angew. Chem.*, 1894, 47) for estimating indigotin after extracting indigo-gluten and indigo red by the aid of dilute hydrochloric acid and a mixture of ether and alcohol, gives very good results provided

the instructions are followed exactly. It is advisable, however, to wash the asbestos filter with water strongly acidified with sulphuric acid, to filter the diluted indigo solution through paper, and also as before to titrate by the "cloud" rather than by the colour of the liquid. The two methods then, with the exception of one abnormal specimen of "fine Bengal" indigo (which it appeared had been made by the patent "Coventry process"), gave concordant figures, lying well within the limits of error (0·6 per cent.) quoted by the previous authors.

Comparative dyeing experiments on wool showed that extracted indigo solutions produced pure uniform shades of blue, whereas the non-extracted solutions gave irregular and paler tints. The tests were but analogous to practical experience, for the depth of colour was not strictly proportional to the real amount of indigo present in the baths. In fact, only similar materials, *i.e.*, such as have been freed from red and gluten, or such as have not been so freed, can fairly be contrasted by dyeing methods; and only extracted indigos give trustworthy indications (within 2 per cent.) as to their actual content in indigo blue. Colorimetric trials were also carried out in Grosse's polarizing colorimeter, and emphasized the necessity for similarity between the samples. Two specimens of indigo, containing 64·9 and 62·7 per cent. of indigotin, used as standards as against two others containing by titration 45·43 and 58·3 per cent., made the latter to come out at 46·02 and 57·96 per cent.; but another pair, which volumetrically contained 46·38 and 47·4 per cent. by comparison with 48·9 and 59·1 per cent. standards, gave only 41·06 and 42·35 per cent. respectively, *i.e.*, 5 per cent. too little, presumably because the shades were divergent. (For full details respecting these dyeing and optical investigations and methods of manipulating the latter apparatus, the original communication must be consulted.)

An earlier process for estimating indigotin by a Kjeldahl nitrogen determination, after the glutinous matter, red, and brown, had been extracted with hot dilute hydrochloric acid, alcohol, and hot dilute potash, was outlined by Voeller (*Zeits. angew. Chem.*, 1891, 110). The idea is only feasible for high grade indigos, (1) because excessive handling with solvents may lead to the formation of isomeric bodies, (2) because lower quality samples contain other nitrogenous substances which are not capable of extraction. Nevertheless, as it has lately been shown that indigo red is a most important body, and serves to fix the blue on the fibre, it might be advantageous to submit the residue of the alcoholic (or ethereal) extract to a nitrogen estimation in order to determine approximately the proportion of red, for otherwise it is not easy to obtain this constituent in a form suited for weighing. The solutions of the gluten and the brown may be treated similarly, but the potassium or sodium hydroxide employed to dissolve the latter should not exceed 1 per cent. in strength. All these extractive matters may conveniently be calculated on the assumption that, like indigotin itself, they contain 10·687 per cent. of nitrogen.

It might be expected that, after all the isomeric bodies have been extracted, every indigo should give a practically identical result by sulphonation and titration with permanganate for the indigotin in it. This is not so, for further changes occur in the material during the treatment with alkali and the subsequent drying at 100° C. It follows, therefore, that when the nitrogenous non-indigo substances have been completely removed, the nitrogen content of the residue corresponds to the amount

of indigotin; but a titration of the extracted matters in no way represents the indigo blue in the original sample.

Copious tables recording the analysis of various indigos by the different processes mentioned above are appended. F. H. L.

Furcora Gigantea, a New Commercial Fibre. A. Herzog. (*Chem. Zeit.*, 1899, xxiii., 314.)—This new fibre is being introduced into the arts as a substitute for manilla, which it approaches closely in tensile strength. It is derived from a species of aloe, recently taken into cultivation in the neighbourhood of Shenstone, Natal.



FIG. 1.



FIG. 2.

The single fibres average 3 millimetres in length and 0·032 millimetres in diameter; the simple cells vary between 1·3 and 6 millimetres long by 0·021 and 0·042 millimetres wide. The general microscopic appearance of the substance is shown in the annexed drawings, Fig. 1 being a longitudinal section, Fig. 2 a transverse section; both are magnified 100 diameters.

Iodine and sulphuric acid colour the fibre yellow. Its methyl number is 9·29, corresponding to 17·56 per cent. of lignin in the dry material, a value which is practically identical with that found in the commoner *Aloe perfoliata* (17·22 per cent.). The ash of the fibre is poor in silica (3·78 per cent. of SiO_2) but very rich in lime. The average proportion of ash is 1·3 per cent. F. H. L.

INORGANIC ANALYSIS.

The Volumetric Determination of Bismuth. C. Reichard. (*Zeit. anal. Chem.*, 1899, xxxviii., 100, 101.)—A weighed quantity of the substance containing the bismuth compound is dissolved in acid, and an excess of alkali added to the solution. The bismuth is then oxidized by means of chlorine water, or, preferably, by passing a current of chlorine through the heated liquid. The precipitate is boiled until its colour changes from orange to dark red. The bismuthic acid is washed by decantation with water to remove the excess of alkali and chlorine, after which a sufficient quantity of a standard solution of arsenious acid in sodium hydroxide is added and the liquid boiled, until the red bismuthic acid is converted into the white hydroxide ($Bi_2(OH)_6$). When the reaction is complete the liquid is rendered strongly acid with sulphuric acid, filtered while hot from the precipitate, and the unoxidized arsenious acid in the filtrate determined by titration with permanganate.

It is advisable to use solutions containing only small quantities of bismuth in

order to shorten the time required for the reduction. A suitable strength mentioned for the arsenious acid solution is about 0·01 gramme of As_2O_3 in 1 c.c.

C. A. M.

Determination of Zinc in Ores containing Alumina. F. Jensch. (*Zeits. angew. Chem.*, 1899, 465.)—In the analysis of zinc ores it is often noticed that the precipitate produced by ammonia carries down with it notable quantities of zinc, and thus consists of a varying mixture of the oxides of iron, aluminium, zinc, and manganese. The author has investigated a number of different minerals to see whether it is the manganese or the alumina which causes this deposition, and he finds that the action is due chiefly to the latter. In most cases, however, a second, and in all cases a third, reprecipitation with ammonia suffices to obtain the whole of the zinc in the united filtrates.

F. H. L.

Determination of Ammonia, Nitrates, and Nitrites in Water. L. W. Winkler. (*Chem. Zeit.*, 1899, xxiii., 454.)—It is one of the inconveniences of the Nessler process for estimating ammonia in water that the tint produced by the sample under examination cannot be matched by running standard ammonium chloride solution into a mixture of ammonia-free water and the Nessler reagent. In the presence, however, of Rochelle salt this becomes possible, for instead of the usual turbidity a clear colour is formed, which lends itself properly to optical inspection. In two similar stoppered cylinders are placed 100 c.c. of the water to be analysed and 100 c.c. of ammonia-free water respectively; 2 or 3 c.c. of a cold-saturated solution of Rochelle salt are added to each, and then the same volume of Nessler's reagent. The cylinders are shaken, and standard ammonium chloride is dropped into the second from a small finely-graduated burette until the tints are identical. Turbid waters should be filtered, rejecting the first portions of the filtrate.

For the estimation of nitrates plus nitrites, two 50 c.c. flasks are taken and charged, one with 10 c.c. of the sample, the other with 10 c.c. of distilled water. To each are added 1 c.c. of a 2 per cent. solution of brucine sulphate and 20 c.c. of sulphuric acid. A solution containing 0·187 gramme of pure potassium nitrate per litre, of which each c.c. is equivalent to 0·1 milligramme of N_2O_5, is dropped into the standard flask till the yellow colours match. If the proportion of nitrates is very small, the quantities of water may be raised to 50 c.c., the other liquids being increased *pari passu*, and to insure great accuracy, the volumes in both flasks should be made identical before comparison. The organic matter in natural waters does not appear to affect the process, for when a bad spring water was boiled with acid permanganate (the excess being destroyed with oxalic acid) the results were the same. Ferrous salts must first be oxidized with permanganate. The brucine reaction indicates nitrites as well as nitrates; the former are determined as follows: 100 c.c. of water are filtered if necessary and freed from ferric iron (ferrous iron is of course immaterial) by treatment with sodium hydroxide, and brought into a 200 c.c. flask having a long narrow neck. Twenty c.c. of 10 per cent. hydrochloric acid and 2 or 3 c.c. of starch solution are added, and then 4 grammes of potassium bicarbonate are dropped in gramme by gramme in order to yield a current of carbon dioxide,

which serves to remove all oxygen from the water and from the flask. A small crystal of potassium iodide and 1 more gramme of the bicarbonate are introduced, and the free iodine is titrated with a solution of thiosulphate made by diluting 26·3 c.c. of he normal reagent to 1 litre (1 c.c. equals 0·1 milligramme of N_2O_3). The results of all three processes are satisfactory. F. H. L.

A Method for Facilitating the Colour Comparison in the Determination of Nitrates in Water by the Phenol-sulphonic Acid Method. F. X. Moerk. (*Amer. Journ. Pharm.*, 1899, lxxi., 157-161.)—The only drawback to this method is the difficulty sometimes experienced in matching the colour, owing to bye-colours being produced by other substances in the water. A correction can be made for these, however, by taking a duplicate portion of the sample of water and volatilizing the nitric acid by heating the residue on the water-bath with ordinary sulphuric acid. In the test proper with phenol-sulphonic acid the organic matter is acted upon as in the duplicate test, while the nitric acid forms a nitro-derivative. Two solutions are thus obtained, one representing only the colour derived from the organic matter, while the colour in the other is due to both organic matter and nitrates. These solutions are suitably diluted, and a sufficient quantity of the standard nitrate solution added to the duplicate to match the colour.

With reference to the determination of ammonia, the author states that the standard ammonium chloride solution gradually deteriorates, and should be re-standardized or replaced from time to time. C. A. M.

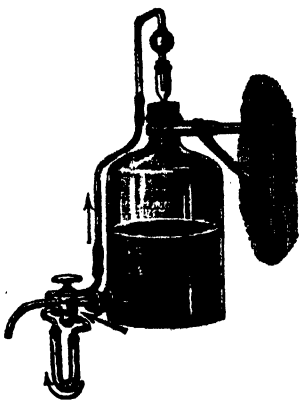
Atomic Weights for Industrial Purposes. H. Erdmann. (*Zeit. angew. Chem.*, 1899, 424.)—This article is essentially an attack on the Commission of the German Chemical Society for taking O = 16 as the unit in their new table of atomic weights (this vol., p. 83), and on W. Fresenius for recently retaining the units H = 1 and O = 16. There are various advantages derived from adopting H = 1 as the basis: all electro-chemical and weight-to-volume calculations are greatly simplified by reason of the fact that 1 litre of hydrogen at normal temperature and pressure weighs 0·0900 gramme; the atomic weights of silver and iodine become round numbers (107 and 126 respectively), and these are two of the substances most frequently determined. The employment of O = 16 destroys these conveniences, while this element is nearly always estimated by difference. The argument that the O = 16 system gives the approximate values C = 12 and N = 14 (instead of 11·9 and 13·9) is vitiated by the unwieldy number of hydrogen = 1·0075. Erdmann would like to see several years elapse with further accurate determination of the true atomic weights before any list claiming international adoption is finally drawn up. F. H. L.

APPARATUS.

A Laboratory Ball-mill. H. Thiele. (*Zeits. angew. Chem.*, 1899, 597.)—The chief feature of this apparatus is in the device employed for rotating the mill. A horizontal, rectangular metal tray, fastened to the wall, contains two parallel lengths of gas-barrel covered with a wooden casing, which serve as supports and driving-gear

for the mill proper. One length runs in fixed bearings, and carries a pulley at the end, or is otherwise connected with the prime motor. The second revolves free, and can be dropped into slots at different distances from its neighbour, so as to accommodate mills of varying sizes. Stops are fixed at the ends of the tray in order to prevent the mill working its way over the ends of the rollers. Any convenient cylindrical vessel can be used as a mill, preferably one made of porcelain; while two or more of slightly different diameters can be revolved simultaneously. F. H. L.

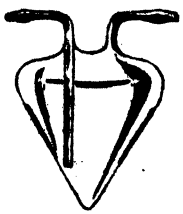
Storage Vessel for Delicate Volumetric Solutions. W. Steinfels. (Reprint from *Zeits. anal. Chem.*; through *Chem. Zeit. Rep.*, 1899, 146.)—The object of this apparatus is to prevent continual access of air and moisture to the protective material placed at the inlet of vessels containing unstable volumetric solutions, so that it may remain efficient for a greater length of time. The essential feature consists in a stopcock with three passages drilled in such positions that the liquid exit, the air entry, and the apertures of the guard tube are either all closed, or, after rotating it through 90°, are all simultaneously open. The sketch illustrates the general arrangement, and the arrows indicate the passage of air into the vessel. For alkaline liquids, the glass cock is replaced by three rubber tubes, controlled by a special "eccentric" clip, which acts in a similar manner. The bottle is slung by the neck, so that it can be shaken, and the bulb tube at the top serves to return any volatilized water.

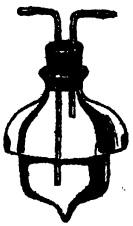


F. H. L.

Some New Apparatus. H. Göckel. (*Zeits. angew. Chem.*, 1899, 494.)

Reflux Condenser for use with Beakers.—The object of this device is to enable an ordinary beaker to be employed for cohobating liquids, instead of the usual flask with vertical tubular condenser. The advantages are various: there is no cork or rubber to be attacked; the solid residue (after distillation) can be removed from the beaker with a spatula, and any volatilized solid matter can be scraped off the external surface of the condenser. As shown in the drawings, the apparatus is made in two forms; one fits a beaker of any size by virtue of its conical shape, the other rests on the top by means of its shoulder. Grooves are provided to permit entry and exit of air when lipless beakers are adopted.



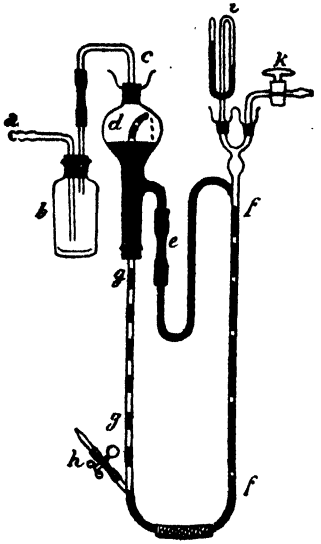


Funnels for use with Filter-plates.—When filter-plates are employed with the pump in an ordinary funnel, they are very liable, especially if at all large in diameter, to slip out of position and so to dislodge the filter-paper. The new funnels accordingly are constructed with horizontal internal grooves against which the edges of the plate (or plates) have a bearing. Thus it is possible to put two or three plates of different sizes in one funnel, filtering the liquid two or three times in one operation.

Convenient media such as sand, glass-wool, etc., can also be packed between the plates when disposed in this fashion.

[All these articles are to be procured from Alt, Eberhardt, and Jäger, of Ilmenau.]

F. H. L.



An Automatic Mercury Pump. F. Friedrichs. (*Zeits. angew. Chem.*, 1899, 498.)—This apparatus has been designed to work with the minimum of mercury, and to be as simple and cheap as possible for ordinary laboratory work. The parts are connected with rubber tubes to make it easier to clean, and the rubber corks are provided with mercury seals. The flask *b* serves to catch any liquid that may flow back from the water-pump attached to *a*, and the manometer is intended as a check on the working of the apparatus rather than as a means of measuring the vacuum. Two clamps suffice to support it securely.

F. H. L.

REVIEW.

ANLEITUNG ZUR DARSTELLUNG CHEMISCHER PRÄPARATE. By Professor Dr. H. ERDMANN. Second edition. (Bechhold, Frankfurt.)

This is an excellent little book, giving explicit and at the same time concise directions for the preparation of typical inorganic substances, intended for the use of students. A good many books have appeared during the last few years dealing with organic preparations, while inorganic ones have been lamentably neglected. In the whole field of inorganic chemistry there is at the present time happily a revival of activity, and the same considerations which have led to the marvellous development of organic chemistry are now yielding copious results in inorganic chemistry.

The preparations chosen by Professor Erdmann cover a very wide field, and embrace preparations of compounds of thirty-five elements. It may safely be said that the student who has worked through this little work will have a wider and more useful knowledge of inorganic chemistry than he could possibly obtain by the usual course of "test-tubing."

Many of the preparations are chosen in view of their application to organic synthesis. Some of them are not hackneyed preparations, but are quite modern, the reduction even of manganese dioxide to fused metallic manganese by means of the Goldschmidt-Vautin reaction, which only dates back to 1898, being included.

O. H.

THE ANALYST.

SEPTEMBER, 1899.

OBITUARY NOTICES.

THE LATE SIR EDWARD FRANKLAND.

It is with deep regret that we have to record the death, full of years and honours, of one of the most eminent of our Honorary Members, Sir Edward Frankland, which took place on August 9, 1899. The deceased was born at Churchtown, near Lancaster, in 1825, and received his general education at the Lancaster Grammar School. He commenced his chemical career as a student under Hofmann at the Royal School of Mines, and afterwards studied in the laboratories of Giessen and Marburg under Liebig and Bunsen respectively. When quite a young man Dr. Frankland commenced that series of researches which was finally destined to yield such important results in the development of the theory of chemistry. These were at first of an analytical nature, and were aimed at the isolation and identification of the proximate radicles contained in certain organic bodies; afterwards problems of a synthetical nature were attacked; these consisted of the building up of organic bodies, and resulted in the discovery, in 1850, of the organo-metallic compounds of methyl and ethyl with zinc. The demonstration that such bodies could be produced was of the greatest theoretical value, since the study of their formation led to certain deductions being drawn having reference to the equivalence of the elements. These, which were communicated to the Royal Society in 1852, have had a vast influence on the course of chemical theory. In 1851 Dr. Frankland was appointed Professor of Chemistry at Owens College, Manchester, and it was there that his first work in relation to applied chemistry was carried out. It consisted in an investigation into the composition of the gases evolved by the various kinds of coal employed in the manufacture of illuminating gas. In 1865 he succeeded his former teacher, Hofmann, at the Royal School of Mines; and also undertook the continuance of the monthly analyses of the waters supplied to London, which had been commenced some months previously by Hofmann for the Registrar-General. Frankland, dissatisfied with the processes then in general use for this purpose, elaborated in conjunction with Armstrong the well-known process which bears their conjoint names. Dr. Frankland was appointed, in 1868, a member of the second Royal Commission on the Pollution of Rivers, which sat for six years, in the course of which a large amount of chemical work was performed that led to a more complete knowledge of the properties of the waters derived from the various water-bearing strata of this

country. In one of his experimental investigations he spent the night in company with Tyndall on the summit of Mont Blanc, determining the rate of combustion of stearin candles in order to discover if this was dependent on the density of the atmosphere. In conjunction with Fick and Wislicenus, Dr. Frankland made a series of determinations of the amount of nitrogen excreted by the human body during severe exercise; the interpretation of these demonstrated that all the force evolved could not be derived from the waste of muscular tissue alone, but must be obtained from the combustion of fatty and allied bodies. In addition to holding the official positions which have been already mentioned, Dr. Frankland was also Lecturer on Chemistry at St. Bartholomew's Hospital, Professor of Chemistry at the Royal Institution (where he succeeded Faraday), and at the Royal College of Science, from which he retired in 1885. He was elected a Fellow of the Royal Society in 1853, and was the recipient of a gold medal from that Society in 1857. The University of Oxford conferred upon him the degree of D.C.L. in 1873, and that of Edinburgh the degree of LL.D. in 1884. Dr. Frankland was President of the Chemical Society in 1871, and was also the first President, in 1877, of the Institute of Chemistry. His merits were fully recognised abroad, for he was either member or corresponding member of numerous foreign academies of science.

THE LATE PROFESSOR ROBERT WILHELM BUNSEN.

It is also with sincere regret that we announce the death, on August 18, 1899, at Heidelberg, of the world-renowned chemist Professor Bunsen, who had reached the mature age of eighty-eight years. Born at Göttingen, he studied science at the University of his native town, taking his degree of Ph.D. there in 1831, and afterwards continued his studies at Paris, Berlin, and Vienna. In 1836 he succeeded Wöhler as Professor of Chemistry at Cassel, and became Assistant Professor at the University of Marburg in 1838. In 1852, after a short interval at Breslau, Bunsen was appointed to the Chair of Experimental Chemistry at Heidelberg, and here, though he received a tempting invitation to Berlin, he spent the remainder of his active career, resigning his professorship in 1889.

Bunsen was an indefatigable investigator and a prolific inventor, several of his devices being of an eminently utilitarian nature. Amongst these may be mentioned the Bunsen battery and the Bunsen burner, the latter of which is now an almost universal household appliance. The truly marvellous discovery, in connection with Kirchhoff, of spectrum analysis, forming as it does a separate and distinct branch of chemical science, will most of all serve to keep his memory in remembrance. Amongst his numerous investigations were those on the explosive compounds of arsenic, an accident during which nearly cost him his life; those, in conjunction with the late Lord Playfair, into the chemistry of the gases evolved by blast furnaces; and those with Sir Henry Roscoe on actinometry and on the chemical action of light. As the result of a visit to Iceland in 1847, came the investigation into the geological conditions of that island and his theory of geysers. But that portion of his labours which appeals most directly to analytical chemists is the working out of his system

of gas analysis, which is so distinguished by the precision of its results that it may be said to have placed this branch of analysis on a firm and secure foundation. These are only a few of his many contributions to chemical and physical science, which are recorded in a numerous series of memoirs.

Pre-eminent as a teacher, and ardently devoted to his laboratory and students, Bunsen succeeded in forming at Heidelberg a school of chemistry rivalling that of Liebig at Giessen; whilst his kindly and genial nature endeared him not only to his numerous students, but also to all his colleagues.

Bunsen was the recipient of many orders and decorations, and also of many academical distinctions. Amongst these latter was his election to the fellowship of the Royal Society in 1858, the awarding to him of the Copley Medal of that Society in 1860, and also to himself and Kirchhoff of the Davy Medal in 1877.

In Bunsen we lose the last connecting link between the great German chemists of the past and those of the present time.

PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS.

ON SOME COMPARATIVE ANALYSES OF, AND DIGESTION EXPERIMENTS WITH, WHITE AND WHOLE-MEAL BREADS.

By Otto Rosenheim, Ph.D., and Philip Schidrowitz, Ph.D.

(*Read at the Meeting, June* 7, 1899.)

The distinct progress made in many branches of food technology, due to the substitution of methods based on scientific principles for empirical practices, does not apply to any extent to the large and important bread industry. There has been no lack of proposals in this direction, but owing partly to their impracticable nature, and partly to the conservatism of the industry concerned, they do not appear to have been adopted to any great extent.

One of the most inviting questions in this connection is the improvement of the nutritive value of bread by increasing its percentage of albuminous matter, which is very small as compared with the carbohydrates and other nitrogen-free substances. In this direction many attempts have been made to make use of the relatively large quantity of nitrogenous matter in the husks or outer parts of the cereals concerned, which at present is wasted in the manufacture of white bread, where only the finely-dressed flour obtained from the inner part of the grain is used.

Although whole-meal breads made from whole-grain flour show on analysis a higher percentage of albuminous matter and phosphates than bread made from white flour, digestion experiments* have shown that the nutritive value of these breads is not materially increased, and these results have found practical confirmation in the taste of the public, which has always evinced, and continues to evince, a decided predilection for ordinary white bread. The chief reason for this fact lies probably in

* *Cf.* Rübner, *Zeitschrift f. Biologie*, 19, and Meyer, *id.*, 1871.

the increased percentage of indigestible cellulose and the physical state of the same. It has been pointed out by different investigators (Lehmann,* Goodfellow,† Prausnitz‡) that the degree of fineness of these breads in general leaves much to be desired, and it is probably due to the mechanical irritation set up by the large flakes of indigestible cellulose in the intestinal tract that the peristaltic action is accelerated, thereby preventing the absorption of much matter which, under ordinary circumstances, would be completely assimilated. This increase of peristaltic activity may occasionally be desirable, but the effects aimed at in such cases are medicinal rather than alimentary.

We have recently had an opportunity of making some experiments with a whole-meal bread the chief advantage of which, according to the patentees, consists in the fact that the cellulose is very finely comminuted, the object of this being to avoid the undesirable results alluded to above. We have made a complete analysis of, and some digestion experiments with, this bread, and for purposes of comparison have similarly examined a white bread and a whole-meal bread of ordinary make under identical conditions, and venture to think the results may be of some interest to the members of the Society. The results of the purely analytical examination are set forth in

TABLE I.

Results of Chemical Analysis of Three Breads: I., Ordinary White Bread; II., Ordinary Whole-meal Bread; III., Patent Whole-meal Bread.

	Percentage Composition of					
	Breads as Examined.			Water-free Breads.		
	I.	II.	III.	I.	II.	III.
	Per cent.	Per cent.	Per cent.	Per cent.	Per cent.	Per cent.
Water	39·10	40·18	48·83	—	—	—
Dry substance	60·90	59·82	51·17	100	100	100
Total ash	0·59	1·88	1·12	0·97	3·14	2·18
Phosphoric acid	0·16	0·51	0·43	0·26	0·85	0·84
Soluble matter	4·73	7·54	10·00	7·77	12·60	19·54
Total albumin	8·25	7·87	6·94	13·54	13·16	13·56
Pure albumin	7·34	7·86	5·52	12·05	13·15	10·78
Soluble nitrogenous matter	0·61	0·73	0·68	1·00	1·22	1·32
Starch	38·45	39·18	31·86	63·13	65·49	62·45
Sugar (maltose)	1·19	1·77	0·62	1·95	2·96	1·21
Dextrin	0·84	0·71	0·52	1·38	1·19	1·01
Cellulose	0·24	1·06	0·86	0·39	1·68	1·68
Fat	0·21	0·63	0·34	0·34	1·05	0·66
Acidity (lactic acid)	0·19	0·29	0·38	0·31	0·48	0·74
Loss of water in 15 days	9·23	—	7·60	—	—	—

It will be seen from the above table that as far as the two brown breads are concerned the analytical figures reveal no striking differences. The soluble matter is

* "Archiv. Hygien.," 19 vol., 73. † "The Dietetic Value of Bread," Macmillan, 1892.
‡ "Archiv. Hygien.," 17 vol., 626.

appreciably higher in III., and this bread has also a slight advantage in the total albumin and soluble nitrogen; on the other hand, the ordinary whole-meal (No. II.) contained appreciably more pure albumin (as determined by Stutzer's method) and fat. The quantity of cellulose was, curiously enough, identical, but the quality was very different. After removing starch, sugar, albumin, fat, etc., by the ordinary methods, the cellulose residue of II. was seen to consist of large, thick, non-transparent entire flakes of a brownish colour, whereas the patent bread cellulose was in the form of very thin, straw-coloured transparent flakes, mostly broken up into irregular shapes.

As far as the white bread is concerned, it will be seen that it contained appreciably less ash, phosphoric acid, and soluble matter than the whole-meal varieties, but its nitrogenous constituents (total, pure and soluble albumin) were, practically speaking, equal to those contained in the former.

The Digestion Experiments.—The digestion experiments were all carried out *in vitro*, and although it has often, and we think with justice, been urged that experiments conducted under these artificial conditions are no criterion for forming an opinion as to the actual processes and changes taking place in the body, yet we venture to think that, conducted on purely comparative lines and under absolutely equal conditions, they are likely to afford a valuable clue as to the relative merits of the different substances concerned, and are to a very great extent comparable *inter se*.

The plan of our experiments was to submit the breads to the action of the chief ferments to which they would be exposed during their passage through the body under conditions as similar as possible to those obtaining in natural digestion, and for this purpose we subjected them :—

(*a*) To the salivary digestive process ;

(*b*) To gastric, followed by the pancreatic, digestion ;

(*c*) To the pancreatic digestive process.

It was not thought necessary to make experiments with gastric digestion alone, inasmuch as bread is never digested by the gastric juice alone either in health or disease.

The digestion of bread, broadly speaking, consists, firstly, in the conversion of starch into sugar, and, secondly, in the transformation of insoluble nitrogenous matter into soluble albumoses and peptones. As a measure of the relative digestibility of the breads, we took on the one hand the quantity of starch converted into sugar, and on the other the percentage of soluble albuminoid formed from equal weights of bread in a given time, the results being calculated on the water-free substances. These figures are not strictly comparable *inter se*, owing to the different percentages of starch and nitrogenous matter in the various breads ; and to eliminate this source of error the results were also calculated on the original percentage of starch and nitrogenous matter.

(*a*) *Salivary Digestion.*—We were unable to find any record in literature of work on the action of the salivary ferments on bread, and this fact may perhaps lend some additional interest to the figures obtained. The measure of the digestibility of the breads when exposed to the influence of salivary digestion was the quantity of sugar formed (*a*) in five minutes, (*b*) after half an hour's exposure to the action of mixed saliva.

Method.—Saliva was collected from three observers, mixed, filtered, and then diluted to twice its volume with distilled water. To 2 grammes of each bread were added 30 c.c. distilled water and 30 c.c. of the mixed saliva, and the whole was then incubated at 35° to 40°. At the end of the proper period the mixtures were removed from the incubator, and further digestion was stopped by bringing them briskly to the boil. The sugar was then estimated in each case according to the usual gravimetric method. The results were as follows :

TABLE II.

Salivary Digestion. I., II., III. as in Table I.

	Breads as Examined.			Water-free Breads.		
	I.	II.	III.	I.	II.	III.
	Percent.	Percent.	Percent.	Percent.	Percent.	Percent.
Sugar formed by saliva :						
1. After 5 minutes' digestion ...	6·82	2·62	3·75	11·19	4·38	7·32
2. ,, 30 ,, ,, ...	13·18	5·98	6·94	21·64	9·99	13·56
Total sugar theoretically formable from starch				70·14	72·76	69·38
Percentage of sugar actually formed calculated to theoretical sugar :						
1. After 5 minutes' digestion ...				15·95	6·02	10·55
2. ,, 30 ,, ,, ...				30·85	13·73	19·54

On looking at the above table, it becomes apparent that, as far as salivary digestibility goes, the white bread was far superior to the others, and that the finely comminuted patent bread gave much better results than the ordinary coarse wholemeal bread.

B. Gastro-Pancreatic Digestion.—The measure of digestibility was the quantity of nitrogenous matter converted after ten hours' gastric, followed by six hours' pancreatic, digestion.

Method.—Two grammes of bread were mixed with 10 c.c. of a standard pepsin solution and 50 c.c. of water, incubated at 37° C. for ten hours, filtered, washed, and the residue then submitted to six hours' pancreatic digestion, as described under *C*. Results :

TABLE III.

Gastro-Pancreatic Digestion.

	Breads as Examined.			Water-free Breads.		
	I.	II.	III.	I.	II.	III.
	Percent.	Percent.	Percent.	Percent.	Percent.	Percent.
Nitrogenous matter digested after 10 hours' digestion with gastric juice, 16 hours' with pancreatic juice ...	6.18	4·28	4·80	10·14	7·99	9·38
Percentage of nitrogenous matter digested to total nitrogenous matter ...				74·89	60·71	69·71

Here also we see the decided superiority of the white bread over both the brown varieties, and of the patent bread over the ordinary whole-meal.

C. Pancreatic Digestion.—In this case the measures of digestibility were: (1) The quantity of sugar formed after (*a*) one hour, (*b*) seven hours, and (2) the quantity of nitrogenous matter digested after 11 hours.

(1) *Transformation of Starch.*—The pancreatic diastatic digestions were made upon the same plan as the salivary ones. A well-known standard solution of pancreatic juice was used. Each digestive mixture was made up as follows: Bread, 2 grammes; pancreas solution, 15 c.c.; distilled water, 45 c.c.; and a few drops of a saturated solution of sodium carbonate. These mixtures were left in the incubator at 37° for the necessary period, and further digestion was then stopped as before. While in the incubator the mixtures were from time to time stirred, and their reaction was seen to be alkaline; the sugar was estimated in each case as before. The results are shown in

TABLE IV.

Pancreatic Starch Digestion.

	Breads as Examined.			Water-free Breads.		
	I.	II.	III.	I.	II.	III.
	Percent.	Percent.	Percent.	Percent.	Percent.	Percent.
Sugar formed by pancreatic digestion:						
1. After 1 hour	21·32	8·52	11·55	35·00	14·24	22·57
2. After 7 hours	21·97	11·82	14·08	36·07	19·75	27·51
Percentage of sugar actually formed calculated to theoretical sugar:						
1. After 1 hour				49·90	19·57	32·53
2. After 7 hours				51·42	27·14	39·65

In this case also the white bread proved itself superior to the two others, not only relatively, but absolutely; and the patent bread gave better results than the coarse whole-meal bread. Another point worthy of notice is, that whereas the quantity of sugar formed in the case of the white bread does not materially increase after the first hour, the differences between the one and seven hour digestions as regards the other two breads is most marked. In the body, where the products of digestion are removed from the intestinal tract, it is most probable that the results would be still more favourable to white bread.

(2) *Pancreatic Transformation of Nitrogenous Matter.*—The measure of digestibility was the quantity of nitrogenous matter transformed after eleven hours' digestion.

Method.—Digestive mixtures made similarly to those used in the case of the pancreatic starch digestions were kept at 37° for eleven hours, and at the end of this period boiled, and then filtered through asbestos. After careful washing with distilled water, the nitrogen in the residue was estimated by the usual methods, and calculated to albumin by multiplication with the 6·25 factor; by deducting the figure thus obtained, together with the original soluble albumin from the total original insoluble albumin, the quantity of albumin actually digested was arrived at.

TABLE V.

Pancreatic Transformation of Nitrogenous Matter.

	Breads as Examined.			Water-free Breads.		
	I.	II.	III.	I.	II.	III.
	Percent.	Percent.	Percent.	Percent.	Percent	Percent.
Nitrogenous matter digested after 11 hours of pancreatic digestion... ...	6·55	5·48	5·56	10·75	9·16	10·86
Percentage of nitrogenous matter digested calculated on total nitrogenous matter				79·38	69·61	80·08

In this case it will be seen that the white bread and the patent bread were practically equal, but both considerably superior to the ordinary whole-meal.

Conclusions.—Although we do not desire to draw any sweeping conclusions from the above experiments, owing to their limited number and somewhat restricted character, we feel justified in saying that they tend to bear out the opinion now generally held in scientific circles as to the great superiority of white bread as far as digestibility pure and simple goes. It is usually believed that whole-meal bread contains considerably more nitrogen than the white article, but this was certainly not the case in the breads examined by us; and although they were selected with care, there is sufficient variation in ordinary flours to account for this apparent anomaly. The whole-meal breads certainly contained considerably more ash, phosphoric acid, and soluble matter. The results obtained with the patent brown bread, in which the grain and husk are very finely comminuted, supports the contention that the digestibility and wholesomeness of whole-meal bread can be greatly improved by careful and thorough grinding and disintegration of the cellulose.

DISCUSSION.

THE CHAIRMAN (Mr. Allen) inquired how much of the ash in these breads was due to salt; the ash in bread depended mainly on the quantity of salt employed. The question suggested itself whether one ought not to determine the quantity of starch rendered soluble rather than the quantity converted into sugar; but seeing that, at a specified temperature, the proportions of dextrin and sugar formed were constant, and that they varied with the temperature, the consideration was of secondary importance. It would, however, be interesting to know whether the sugar was calculated as dextrose or maltose. He thought it was quite clear that the duty done in the system by whole-meal bread, apart from its irritant powers, must be represented by the quantity of material actually digested; and although digestion experiments carried out in glass were necessarily of an empirical nature, it was only by proceeding tentatively in this direction that any true knowledge of the matter could be gained.

Dr. J. A. VOELCKER said that, regarding the matter rather from a cattle-feeding point of view, it seemed difficult to believe that the larger proportion of phosphoric acid shown in the whole-meal bread could be without value. In the case of the

patent bread, the question of its mode of preparation naturally arose. It was to be noted, however, that it contained considerably more water than either of the others. For the results of digestion experiments to be comparable, it was necessary that the different materials should be comparable as regards mechanical condition. The authors had attempted to insure this by reducing the whole-meal bread to the same condition of fineness as the others, but whether that was successful was certainly doubtful. He did not think it was possible by merely grinding one material to the same degree of fineness as another, to imitate the mechanical condition of the latter, when it had perhaps been quite differently treated *ab initio*. It seemed curious that so slight a difference as existed between the quantities of starch in No. 1 and No. 2 should be followed by so large a difference in sugar produced as was indicated by the salivary digestion experiment, and he thought it likely that, owing to their necessarily different mechanical composition, the two materials were in reality not being treated on exactly the same level. He agreed with the authors that the superiority of whole-meal bread had been exaggerated in some directions, but he was still open to be convinced that such constituents as the phosphoric acid and the soluble matters were without value, or that there was any reason for the less rapid production of sugar in the whole-meal bread other than that attaching to its different mechanical condition.

Mr. HEHNER said that many actual feeding-experiments were on record concerning the comparative digestibility of bread made of whole-meal and of white flour. 'A list of them was contained in the first volume of König, and they showed that while white bread was almost completely digested, both as regards nitrogenous matters and carbohydrates, brown or black breads gave far less favourable results. These physiological experiments were, therefore, in entire agreement with the artificial digestion experiments of the present authors.

Although the analyses showed that whole-meal flour was somewhat richer in nitrogen than white flour, this was a matter without real significance for the organism, as some of the nitrogen was unavailable to the digestive organs. It appeared to him that the cellulose envelope, which, of course, exercised a protective action over the cell-contents, did not explain the circumstance that the authors had found so low a proportion of starch to be converted into soluble products. It rather appeared as if the bran, with its high proportion of pentosans, exercised not only a protecting action, but also an inhibitive one, upon digestion. In the case of ruminant animals who had the power to digest cellulose and other carbohydrates, whole-meal would no doubt show more favourable results, but in the case of human beings, all evidence appeared to show the superiority of the carefully-prepared white over the whole-meal flour.

Analytical figures, taken by themselves, without either artificial or actual digestion experiments, could not show which of two or more vegetable substances would be the most useful to the organism.

Mr. ARTHUR MARSHALL inquired if the authors knew whether the flour used for the white bread was roller-milled or stone-milled. At least 90 per cent. of the white bread baked in this country was prepared from roller-milled flour, but a few of the best firms still employed stone-milled flour. The latter gives a loaf moister and of far

better flavour than the former. In roller-milled flours the germ of the wheat is entirely removed; consequently they contain a considerably smaller percentage of fats and albuminoids. In making whole-meal bread it is necessary to use a larger proportion both of salt and yeast. This would probably account for the greater percentage of ash and soluble matter shown in the analyses.

Mr. CHAPMAN said that the failure to separate the bran in whole-meal flour resulted in the presence of an active enzyme (cerealin), so that during panification, the conversion of starch into dextrin and sugar went on very rapidly. The bread consequently had a tendency to be soft and sodden, and the percentage of soluble matters in it increased. It was difficult, by the ordinary yeast process, to produce brown bread that did not contain an excessive proportion of water, and in making such bread it was customary to use, instead of yeast, hydrochloric acid and carbonate of soda. This fact might account for the high ash in the author's sample. The high proportion of soluble matter in the patent bread might have been due to the presence of this active enzyme, and to the starch having been in a condition particularly amenable to its action. Whole-meal bread was generally more acid than white bread, for its moist and clammy condition was very favourable to the progress of lactic fermentation.

THE CHAIRMAN (Mr. Allen) observed that for many years the percentage of ash in flour had been gradually decreasing, owing possibly to the increased employment of roller-milling processes. It used to be normally 0·72 per cent., but half this proportion was now very common.

Dr. SCHIDROWITZ said that the sugar in each case was stated in terms of glucose. It was not suggested that the comparatively high percentage of such constituents as phosphoric acid in brown bread was entirely without value, but he personally thought that, except under special circumstances, the question of digestibility was the most important one. He quite agreed that experiments on individuals were far preferable to artificial experiments, though even here the personal factor would have to be considered. He did not think that the excess of soluble matter in the patent bread was very extraordinary, seeing that the chief advantage of this bread was that the cellulose and other ingredients were very finely comminuted, and this fact also accounted for the relatively larger amount of starch converted. He did not actually know the terms of the patent under which it was manufactured, but believed that the whole grain was converted into flour in one operation.

OFFICIAL EXAMINATION OF WOOD-NAPHTHAS FOR METHYLATION PURPOSES.

(*Abridged from the "Pharmaceutical Journal," July* 15 *and August* 12, 1899.)

MAKERS of methylated spirits are, according to a notice just issued by the Commissioners of Inland Revenue, to be informed in future, as precisely as possible, what kind of wood naphtha will be accepted as suitable for methylating purposes, as well as the nature of the objections to any samples which may be rejected. All wood naphtha submitted for approval must consist entirely of substances derived

from the destructive distillation of wood. It must also be so impure as to make nine times its volume of spirit of wine sufficiently nauseous to be rendered incapable of being used as a beverage, or of being mixed with potable spirits of any kind without rendering them unfit for human consumption. Not more than 30 c.c. of the wood naphtha must be required to decolorize a solution containing 0·5 gramme of bromine; it must also be neutral or only slightly alkaline to litmus, and require at least 5 c.c. of decinormal acid to neutralize 25 c.c. of the spirit when methyl-orange is used as the indicator. No wood naphtha will be approved which contains (*a*) less than 72 per cent. by volume of methyl alcohol; (*b*) more than 12 grammes per 100 c.c. of acetone, aldehydes, and higher ketones, determined as "acetone" by the formation of iodoform according to Messinger's method; (*c*) more than 3 grammes per 100 c.c. of esters, determined as methyl acetate by hydrolysis.

We are enabled, through the courtesy of the officials at the Government Laboratory, to give the following details of the tests for wood naphtha:

Bromine Decolorization.

A standard bromine solution is made by dissolving 12·406 grammes of potassium bromide and 3·481 grammes of potassium bromate in a litre of recently boiled distilled water.

50 c.c. of this standard solution (= 0·5 gramme bromide) are placed in a flask of about 200 c.c. capacity, having a well-ground stopper. To this is added 10 c.c. of dilute sulphuric acid (1 in 4) and the whole shaken gently. After standing for a few minutes the wood naphtha is slowly run from a burette into the clear brown solution of bromine until the latter is completely decolorized. Not more than 30 c.c. of the wood naphtha should be required for this purpose.

Methyl-Orange Alkalinity Test.

The naphtha should be faintly acid to phenolphthalein; slightly alkaline or neutral, rarely acid, to litmus, and always alkaline to methyl-orange. 25 c.c. of the wood naphtha are placed in each of two beakers, and titrated with decinormal acid, using in the one case a few drops of litmus solution, and in the other of a solution of methyl-orange as indicator. With litmus usually 0·1 to 0·2 c.c. of decinormal acid is required to neutralize. With methyl-orange the total alkalinity should be greater —at least 5 or 6 c.c. of decinormal acid being required for neutralization.

The total alkalinity, less that given with litmus, is the "methyl-orange alkalinity," and, for the 25 c.c. of wood spirit, should not be less than is required to neutralize 5 c.c. of decinormal acid.

Estimation of Methyl Alcohol.

22 grammes of coarsely powdered iodine and 5 c.c. of distilled water are placed in a small flask and cooled by immersion in ice-cold water. Then 5 c.c. of the wood spirit (60·0 o.p.) are added, the flask corked, the contents gently shaken, and allowed to remain in the ice-cold bath for ten to fifteen minutes.

When well cooled 2 grammes of red phosphorus are added to the mixture of spirit and iodine in the flask, and the latter is immediately attached to a reflux condenser.

The reaction soon commences, and must be moderated by dipping the flask into a cold-water bath. (Spirit may be lost if the reaction is too violent.) After about fifteen to twenty minutes, when all action appears to have ceased, the water-bath under the flask is gradually heated to a temperature of about 75° C. (167° F.), and the flask, being occasionally shaken, is allowed to remain at this temperature for fifteen to twenty minutes. The source of heat is then removed and the apparatus left for an hour till it has cooled, when the condenser is reversed, and the methyl iodide slowly distilled off—first at a low temperature—the bath being allowed to boil towards the end of the operation only. The end of the condenser dips into water in a measuring tube, and the iodide is collected under water and measured at a temperature of 15·5° C. (60° F.).

The percentage (by volume) is found from the formula:

$$\frac{\text{C.c. methyl iodide found} \times 0{\cdot}647 \times 100}{\text{C.c. wood spirit taken.}} = \text{Percentage (by volume) of methyl alcohol.}$$

Or when 5 c.c. of spirit are taken:

$$\text{C.c. methyl iodide} \times 12{\cdot}94 = \text{percentage (by volume).}$$

Esters, acetals, etc., also yield methyl iodide by this process, and from the percentage of methyl alcohol calculated as above an amount equivalent to the percentage of these substances present must be deducted. Practically, however, methyl acetate is the only compound usually found in quantity sufficient to materially affect the result. The grammes of methyl acetate per 100 c.c. of spirit multiplied by 0·5405 give the equivalent of methyl alcohol to be deducted from the total percentage by volume calculated from the methyl iodide found.

The Acetone Reaction.

25 c.c. of normal soda are placed in a flask similar to those used in the bromine reaction. To this is added 0·5 c.c. of the naphtha. The mixture is well shaken, and allowed to stand five to ten minutes. Into it from a burette $\frac{N}{5}$ iodine solution is run slowly, drop by drop, vigorously shaking all the time till the upper portion of the solution, on standing a minute, becomes quite clear. A few c.c. more of $\frac{N}{5}$ iodine solution are added, as to get concordant results an excess of at least 25 per cent. of the iodine required must be added. After shaking, the mixture is allowed to stand for ten to fifteen minutes, and then 25 c.c. normal sulphuric are added. The excess of iodine is liberated, titrated with $\frac{N}{10}$ sodium thiosulphate solution and starch, and half the number of c.c. of thiosulphate solution used are deducted from the total number of c.c. of iodine solution used. The difference gives the amount of acetone by weight in the naphtha by the formula:

$$\text{C.c. } \tfrac{N}{5} \text{ iodine solution required} \times 0{\cdot}3876 = \text{grammes of acetone per 100 c.c. of wood naphtha.}$$

This includes as acetone any aldehydes, etc., capable of yielding iodoform by this reaction.

If the quantity of "acetone" is excessive, a less quantity of the spirit is taken, or 10 c.c. are diluted with 10 c.c. of methyl alcohol free from acetone, and 0·5 c.c. of the mixture is used.

Estimation of Esters.

5 c.c. of the wood naphtha are run into a silver pressure flask of about 150 c.c. capacity, together with 20 c.c. of recently-boiled distilled water. 10 c.c. of normal soda solution are added, the flask securely closed and digested for at least two hours in a water-bath at 100° C. (212° F.). The contents are then washed into a beaker, and titrated with normal acid and phenolphthalein. The difference between the number of c.c. of soda taken and of the acid required for neutralization may be calculated as methyl acetate (weight in volume) from the formula:

$$\frac{0{\cdot}074 \times \text{c.c. soda required} \times 100}{\text{C.c. naphtha taken.}} = \text{grammes per 100 c.c.}$$

Or if 5 c.c. of spirit are taken as above:

1·48 × c.c. soda required = grammes of methyl acetate per 100 c.c. of spirit.

ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

FOODS AND DRUGS ANALYSIS.

A New Test for Cocaine. G. L. Schaefer. (*Amer. Journ. Pharm.*, 1899, lxxi., 222, 223.)—The tests commonly employed for the detection of other coca alkaloids in cocaine salts are the permanganate test for cinnamyl cocaine, and the ammonia (or McLagan's) test for alkaloids which resist the action of permanganate.

As various doubts have been expressed as to the value of the latter test, the author has devised a substitute for it, based on the fact that cocaine chromate is much more soluble in water, or water acidified with hydrochloric acid, than the chromates of the other alkaloids.

The following is recommended as a simple and satisfactory method of determining the purity of the salts of cocaine. In the case of the hydrochloride, for example, 0·05 gramme is dissolved in 20 c.c. of water, and to the solution are added 5 c.c. of a 3 per cent. solution of chromic acid and 5 c.c. of 10 per cent. hydrochloric acid, the temperature of the liquid being kept preferably at 15° C. With pure cocaine hydrochloride the solution will remain clear, but if more than traces of other coca alkaloids be present the liquid becomes cloudy, either immediately or after some time, according to the amount of impurity. C. A. M.

On the Relative Digestibility of certain Food Fats in the Human Alimentary Canal. (*Zeit. für Untersuch. der Nahr. und Genussmittel.*, ii., 484-506.)—This paper contains an account of experiments on digestibility which were made with three descriptions of margarine and a pure Holstein dairy butter. The procedure consisted in feeding a man of normal digestive powers with weighed amounts of the different fats, in addition to a mixed diet embracing beer, coffee, tea, sugar, bread,

lean beef and vegetables, and determining the undigested fat voided in the excreta. The amount of fat consumed per day varied between 112 and 148 grammes. The average results for periods of twenty-four hours were as follows:

Description.	Fat consumed: grammes.	Air-dried Excreta: grammes.	Dry Excreta: grammes.	Fat in Dry Excreta: per cent.	Total Fat: grammes.	Fat absorbed: per cent.
Margarine I.	138·35	47·57	43·08	12·16	4·62	96·68
,, II.	118·64	38·85	35·53	11·60	3·91	96·70
,, III.	112·89	35·01	32·19	10·78	3·46	96·93
Butter ...	111·79	46·29	42·33	11·88	4·82	95·69

In the following table, in which the total quantities for each period are given, regard is taken for the digestive fat consumed in the ordinary diet:

Periods.	Fat Consumed.			Fat Eliminated.					
	Total Fat.			Total Fat.			Undigested Fat.		Fat Absorbed: per cent.
	From Margarine or Butter: grammes.	From the Ordinary Diet: grammes.	Together: grammes.	From Margarine or Butter: grammes.	From the Ordinary Diet: grammes.	Together: grammes.	Of the Total Fat: per cent.	Of the Margarine or Butter: per cent.	
	Margarine-fat.			Margarine-fat.				Margarine-fat.	Margarine-fat.
I. ...	810·65	20·53	831·18	21·45	6·28	27·73	3.32	2·65	97·35
II. ...	459·92	1465	474·57	12·02	3·61	15·63	3·30	2·61	97·39
III. ...	431·88	19·66	451·54	9·05	4·80	13·85	3·07	2·10	97·90
	Butter-fat.			Butter-fat.				Butter-fat.	Butter-fat.
IV. ...	428·16	19·00	447·16	14·87	4·39	19·26	4·31	3·47	96·53

H. H. B. S.

Hydrastine Periodide, and the Assay of Hydrastis Canadensis. **H. M. Gordin and A. B. Prescott.** (*Amer. Journ. Pharm.*, 1899, lxxi., 257-266.)—On adding a solution of iodine in potassium iodide to a solution of a salt of hydrastine a dense brown precipitate is obtained, which approaches a tri-iodide in composition, but appears to be a mixture of different periodides.

When, however, a weak alkaloidal solution is added to a large excess of iodine, a definite compound, hydrastine hydriodide pentiodide, is precipitated as an amorphous brown powder. This dissolves with difficulty in ether, benzene, or cold chloroform, but is more soluble in hot chloroform and alcohol, and dissolves readily in a mixture of alcohol and chloroform or alcohol and ether. When heated in water it fuses to a resinous mass.

The analysis of this periodide gave results corresponding with the formula $C_{21}H_{21}NO_6.HI.I_5$, from which its iodine factor was calculated to be 0·60403.

In the authors' iodometric estimation of hydrastine the details of the process resemble those of the general method described in a former communication (ANALYST, this vol., 74), and the only precaution to be observed is to insure the formation of the periodide by having the alkaloidal solution weak and the iodine in large excess.

In test experiments solutions were prepared containing 0·30 and 0·15 per cent. of hydrastine, and the amounts found by titration with iodine were 0·29 and 0·14 per cent. respectively.

The method worked out by the authors for the estimation of hydrastine and berberine in *Hydrastis Canadensis* is based on the following facts:

1. Hydrastine is soluble in absolute ether, berberine completely insoluble.

2. Berberine hydriodide is nearly insoluble, especially in the presence of a very large excess of potassium iodide.

3. Berberine is precipitated completely on adding to a very dilute solution 10 to 15 times its amount of acetone, and then rendering the liquid strongly alkaline with sodium hydroxide. This berberine acetone is readily decomposed by mineral acids, with the liberation of berberine.

In the assay, 10 grammes of the root are rubbed to a paste with a few c.c. of a mixture of 5 c.c. of ammonium hydroxide ("stronger ammonia"), 5 c.c. of alcohol, and 30 c.c. of ether.

Chloroform, which was used in the general method of extracting alkaloids (ANALYST, this vol., 8 and 74), cannot be employed here, since it combines with berberine, forming a compound not decomposable by acids.

The paste is left in a stoppered jar overnight, is then exposed to a current of air, and when all odour of ammonia has disappeared is kept *in vacuo* over sulphuric acid for five or six hours. The dry powder is introduced into a Soxhlet apparatus, the jar being rinsed out several times with powdered glass or barium nitrate, and is extracted with 40 to 50 c.c. of ether until a few drops of the extract give no reaction with Mayer's or Wagner's reagent.

The ethereal extract is poured into a flat evaporating dish, and the flask washed out several times with water containing about 2 per cent. of sulphuric acid. The washings are added to the main solution, and the dish exposed in a current of air at about 30° C. until the ether has evaporated.

The liquid is then transferred to a 100 c.c. flask and made up to the mark, giving a solution of hydrastine sulphate, of which each 10 c.c. represents 1 gramme of the root. Of this filtered solution 20 c.c. are run from a burette into a flask containing 20 to 30 c.c. of standard iodine solution (about 1 per cent.), and the amount of iodine consumed multiplied by 0·60403 gives the amount of hydrastine.

For a gravimetric estimation a second portion of 20 c.c. is shaken in a separating funnel with benzene and ammonium hydroxide. The colourless benzene layer, which contains the whole of the hydrastine, is transferred to another separating funnel, and shaken with water acidulated with sulphuric acid. Finally the hydrastine is extracted from this aqueous solution by means of ether and ammonium hydroxide, and the solution allowed to evaporate in a weighed beaker in a dark place, dried over sulphuric acid *in vacuo*, and weighed. The hydrastine is left in white crystals with a slight yellowish tint on the side of the beaker, which the authors attribute to the presence of traces of canadine.

In order to determine the berberine, the ether is removed from the Soxhlet extractor by means of a current of air, and the extraction continued with 40 to 50 c.c. of alcohol until the extract becomes colourless.

The alcoholic solution of berberine and extractive matters is transferred to an evaporating dish (the flask being washed with hot water containing a little acetic acid), and kept on the boiling water-bath, whilst water is added from time to time until the alcohol has been evaporated. A little more dilute acetic acid is now added, the dish covered, and the contents, when cold, filtered into an Erlenmeyer flask holding from 300 to 400 c.c.

From 6 to 8 c.c. of acetone are next introduced, followed by a 10 per cent. solution of sodium hydroxide, which is added drop by drop until the precipitate ceases to disappear on shaking, and the liquid becomes strongly alkaline. The flask is then closed, shaken in a circular direction for from ten to fifteen minutes, and allowed to stand in a cool place for three or four hours.

The supernatant liquid is poured on to a small filter, and the crystalline berberine acetone washed first by decantation and then on the filter until the washings are colourless. The filter is then pierced, and the precipitate returned to the flask, into which are also introduced 4 or 5 c.c. of 5 per cent. sulphuric acid, and sufficient water to make about 100 or 200 c.c. in all. When the flask is immersed in boiling water the precipitate is rapidly dissolved. The resulting solution is transferred to a long-necked flask, which is placed on a hot asbestos plate so that the liquid is kept gently boiling for an hour and a half or two hours, hot water being added from time to time if required.

After this the liquid is cooled, poured into a litre flask containing 100 c.c. of a $\frac{N}{20}$ solution of potassium iodide, made up to the mark, and allowed to stand overnight. The next day 500 c.c. of the liquid are filtered into another flask, and after the addition of 50 c.c. of $\frac{N}{20}$ silver nitrate and nitric acid, made up to a litre and filtered. Five hundred c.c. of this filtrate are titrated back with $\frac{N}{10}$ ammonium thiocyanate, ferric ammonium sulphate being used as indicator. Twice the number of c.c. of the thiocyanate solution used is equivalent to the number of c.c. consumed by the berberine, representing 10 grammes of the root. The amount of potassium iodide multiplied by the factor 0·167125 gives the percentage of anhydrous berberine in the root.

The results of the analysis of three samples of *Hydrastis Canadensis* by this method are shown below:

	Hydrastine.		Anhydrous Berberine.
	Iodometric.	Gravimetric.	
No. 1	2·29	2·29	2·52
No. 2	2·33	2·30	2·55
No. 3	2·35	2·28	2·47

With reference to the other *Hydrastis* alkaloid, canadine, the authors state that its proportion is too small to affect the results obtained in the determination of hydrastine or berberine. They consider it probable that the small traces present adhere to one or other of the other alkaloidal precipitates, for they have proved that no appreciable quantity of alkaloid remains in the root after the two extractions.

C. A. M.

Some Colour Reactions of Morphine and its Derivatives. R. Kobert. (*Apoth. Zeit.*, 1899, xiv., 259; through *Chem. Zeit. Rep.*, 1899, 158.)—With a mixture of 2 or 3 drops of formalin and 3 c.c. of strong sulphuric acid, morphine and its derivatives give the following visual and spectroscopic colour reactions. Morphine itself becomes purple-red, then violet, bluish-violet, and finally pure blue; in the spectroscope orange and yellow are suppressed. Dionine (ANALYST, 1899, xxiv., 128) turns dark blue, and in a narrow tube remains so for many hours; in the spectroscope it absorbs orange and yellow, but not so completely as morphine. Codeine or its phosphate, gives a reddish-violet changing to blue-violet; spectroscopically it also absorbs orange and yellow. Heroin (diacetyl ester of morphine), as base, behaves like codeine. Peronine (hydrochloride of benzylic ester of morphine) remains reddish-violet for several hours, and absorbs all light beyond and including yellow. Methyl-phenomorpholine, or its hydrochloride, becomes and remains intensely red; in the spectroscope it absorbs the yellow and green and part of the orange rays.

F. H. L.

Adulteration of Scammony with Galena and Starch. F. Baucher. (*Ann. de Chim. Anal.*, 1899, iv., 186, 187.)—A sample of scammony recently submitted to the author for analysis was regarded as suspicious from the presence of a number of small holes containing well-defined bluish-gray metallic crystals. On extracting the finely-powdered substance with ether, only 41·3 per cent. of resin was obtained, as against 75 to 80 per cent. yielded by commercial scammony of good quality.

The ash left on incineration amounted to 16·6 per cent., and had a reddish-orange colour, whereas the pure substance only yields from 3 to 8 per cent. of white or grayish ash.

The residue insoluble in ether contained the gray metallic crystals, which were identified as galena, and also a large amount of starch. C. A. M.

The Assay of Belladonna Leaves. F. X. Moerk. (*Amer. Journ. Pharm.*, 1899, lxxi., 320-326.)—Since the publication of his former paper (ANALYST, xxiv., 208) the author has made experiments with the object of improving the process therein described. He finds that the extraction of the leaves can be effected more rapidly and completely by means of a solvent consisting of 90 parts by weight of 95 per cent. alcohol with 10 parts of 10 per cent. ammonium hydroxide than by his former solvent.

The following method of working is now recommended: Twenty grammes of the powdered belladonna leaves are shaken at intervals, for from thirty minutes to an hour with 96·5 grammes of the solvent in a stoppered bottle, the liquid is then filtered into a dry flask, care being taken to avoid evaporation as far as possible. The flask and its contents are weighed, the solution transferred to an evaporating basin containing a small glass rod, and its weight found by weighing the empty flask.

The liquid is evaporated on the water-bath at 50° C., until the extract can no longer be stirred with the rod, and its weight now gives the amount of extract.

It is then washed into a separating funnel by means of successive small portions (1 to 2 c.c.) of a mixture of 0·5 c.c. of ammonium hydroxide and 10 c.c. of water,

and is extracted with a mixture of ether and chloroform. The remainder of the process is essentially the same as that described in the previous paper.

Some of the results thus obtained as compared with those given by the original method are shown in the following table:

	Modified Process.		Original Process.	
	Moisture. Per cent.	Alkaloid. Per cent.	Moisture. Per cent.	Alkaloid. Per cent.
Powdered leaves, B ...	9·15	{0·4944 0·4997	9·15	{0·4839 0·4891
English, cultivated ...	8·85	0·6312	8·40	{0·5996 0·6040
German, cultivated ...	8·43	0·5155	7·60	{0·5155 0·5050
German, wild, I. ...	8·50	—	6·80	{0·4997 0·4997
,, ,, II. ...	8·60	0·4208		
Powdered leaves, K ...	7·60	0·3629		

C. A. M.

ORGANIC ANALYSIS.

The Valuation of Saccharin. E. E. Reid. (*Amer. Chem. Journ.*, 1899, xxi., 461-471.)—Remsen and Burton (*Amer. Chem. Journ.*, xi., 403) showed that benzoic sulphimide, when boiled with dilute acids, is converted first into *o*-sulphamine-benzoic acid, and then into the ammonium salt of *o*-sulphobenzoic acid, while *p*-sulphamine-benzoic acid is not affected. Hefelmann (*Ph. Cent.*, lxxxv., 107, and 1895, No. 16) has described a method of estimating benzoic sulphimide in which it is hydrolysed in this way by 71 per cent. sulphuric acid, and the ammonia produced liberated with alkali and distilled. The author has examined this method in comparison with the results obtained by hydrolysis with hydrochloric acid. As 1 c.c. of his standard acid was equivalent to 26 milligrammes of sulphimide, 26 × 25, or 650, milligrammes of the sample were taken for each analysis, so that the number of c.c. of acid neutralized by the ammonia multiplied by four gave directly the percentage of benzoic sulphimide.

In each case 0·650 gramme was mixed in a test-tube with 10 c.c. of 71 per cent. sulphuric acid, prepared by mixing 132 grammes of the pure concentrated acid with 37 c.c. of water. The test-tube was loosely closed by means of a cork, through which passed a glass rod, bent into a loop at the end, with which the mixture was frequently stirred during the heating. After being kept in the boiling water-bath for three hours, the contents of the tube were allowed to cool, diluted with water, distilled, after the addition of 15 grammes of sodium hydroxide, and the ammonia received in standard acid.

The following is a summary of the results in percentages of benzoic sulphimide thus obtained: Commercial saccharin, A, 54·8 to 55·8; commercial saccharin, B, 94·0 to 94·1; mixture of pure para-sulphamine-benzoic acid, and pure sulphimide (86·8 per cent.), C, 86·7 to 88·0; mixture containing 72·7 per cent. of benzoic sulphimide, D, 72·9 to 73·6; commercial saccharin, E, 98·5 to 99·1; commercial saccharin, F, 88·6 to 89·4; pure benzoic sulphimide from (? crystallised) acetone, G, 98·9 to 99·7; average, 99·2.

As most of the results with mixtures of benzoic sulphimide with the para-acid

gave results slightly too high, a number of determinations were made with a pure specimen of the latter, in which it was found that on the average 0·300 gramme yielded 0·51 milligramme of ammonia, equivalent to 5·9 milligrammes of para-sulphamine-benzoic acid. From this the author concluded that the para-acid was slightly hydrolysed by 71 per cent. sulphuric acid at 100° C. When the mixture was overheated, which happened on several occasions, through the water-bath becoming dry, the hydrolysis was considerable, and in one case amounted to 86 per cent. of the acid present.

For the hydrolysis with hydrochloric acid the following method was devised: 0·650 gramme of the saccharin is mixed in a 100 c.c. flask with 50 c.c. of dilute hydrochloric acid (120 c.c. of concentrated acid, diluted to a litre). The flask is closed with a cork, through which passes a glass tube 45 cm. long and 8 mm. wide, placed on a sand-bath, and its contents allowed to boil gently for two hours. The stopper is then removed, and the liquid evaporated to about 10 c.c., after which it is diluted, washed into the distilling flask, 20 c.c. of a solution containing 0·5 gramme of sodium hydroxide in 1 c.c. added, the ammonia distilled into standard acid, and the excess of the latter titrated back, with cochineal as indicator. Acid about $\frac{1}{4}$ N in strength is said to be the most convenient.

In the tabulated results obtained by this method the average with pure benzoic sulphimide was just 1 per cent. higher than that obtained with a similar sample in the sulphuric acid method, whilst the results of the analyses of test mixtures of sulphimide and para-sulphamine-benzoic acid were on the average 1 per cent. lower—a fact which the author attributes to the conversion of some of the latter by the sulphuric acid. In the analysis of samples of commercial saccharin the average results were 0·8 per cent. higher than those given by the same samples when hydrolysed with sulphuric acid.

Experiments made with pure para-sulphamine-benzoic acid showed that, under the conditions described above, it was not attacked by hydrochloric acid.

Benzoic sulphimide is readily converted into the sodium salt of *o*-sulphamine-benzoic acid by being boiled with sodium hydroxide solution, but is not broken down by the treatment. For instance, 650 milligrammes of saccharin E, when boiled with 75 to 100 c.c. of water, containing 10 grammes of sodium hydroxide, yielded only 0·05 milligramme of ammonia. On now adding an excess of hydrochloric acid to residue in the distilling flask, boiling the liquid for several hours, evaporating down, and then distilling with sodium hydroxide, an amount of ammonia corresponding to 98·5 per cent. of sulphimide was found.

From this it appears that benzoic sulphimide may be estimated in the presence of ammonium salts, or salts of other volatile bases; of benzamide, or amides of fatty acids; or, in fact, of any nitrogenous body which is readily broken down by boiling with a 10 to 15 per cent. solution of sodium hydroxide.

The following experiment is described to show the possibility of separating in this manner different classes of nitrogenous organic compounds. A mixture was made containing 0·1478 gramme of ammonium chloride, 0·5000 gramme of *p*-nitro-benzamide, 0·6500 gramme of benzoic sulphimide, and 0·3750 gramme of *p*-sulphamine-benzoic acid.

On distillation with 4 grammes of magnesium chloride and 0·5 gramme of sodium hydroxide, 47·9 milligrammes of ammonia were found, as against 47·1 milligrammes, the calculated amount in the ammonium chloride taken.

The residue in the distilling flask was mixed with 5 grammes of sodium hydroxide, and again distilled. The ammonia found amounted to 51·1 milligrammes, corresponding with 499 out of 500 milligrammes of *p*-nitrobenzamide.

The contents of the flask were then neutralized with hydrochloric acid, about 8 c.c. of the concentrated acid added, and the whole boiled for several hours and concentrated by evaporation. The ammonia obtained on distilling with sodium hydroxide corresponded to 99·1 per cent. of the saccharin present. An excess of sulphuric acid was added to the residue, and the mixture heated. On now distilling with sodium hydroxide, 36·2 milligrammes of ammonia were obtained, instead of the theoretical 33·7 milligrammes representing the *p*-sulphamine-benzoic acid. This high result was due to inadvertent overheating with the sulphuric acid, with the result that not only the para-acid had been broken down, but also some of the *p*-nitro-benzoic acid. Otherwise, it would finally have been possible to determine the nitrogen in the nitro-group by Kjeldahl's process.

In the analysis of pure para-sulphamine-benzoic acid heating with pure sulphuric acid alone at 230° to 260° C. is sufficient to produce complete hydrolysis. In the author's opinion, the decomposition can probably be effected at a lower temperature, and it is suggested that a means of determining *p*-sulphamine-benzoic acid in the presence of other nitrogenous bodies may be based on this property.

C. A. M.

The Occurrence of Estragol in the Essential Oil of Chervil. E. Charabot and L. Pillet. (*Bull. Soc. Chim.*, 1899, xxi., 368-370.)—Grimaux first discovered estragol, the isomer of anethol, in estragon oil (*Comptes Rendus*, cxvii., 1189), and it has since been found by different observers in various essential oils, including those of anise and basil.

The authors have obtained from 36 kilos of the fresh seeds of chervil (*Chærophyllium sativum*) 4·25 grammes of a clear yellow essential oil, with an odour recalling that of anise and estragon. On standing, a few crystals separated out, which were found to consist of a paraffin analogous to those found in a number of essential oils. The main constituent of the oil was estragol (paramethoxy-allyl-benzene), which was identified by converting it into anethol (paramethoxy-propenyl-benzene), and oxidizing the latter to anisic aldehyde. The authors point out that the result of their investigation adds another to the long list of essential oils containing phenols or phenol esters with side allyl chains [$R - CH_2 - CH = CH_2$].

These are of much more frequent occurrence in essential oils than are the isomeric compounds with side propenyl chains [$R - CH = CH - CH_3$] of which the only representatives known to occur naturally are anethol (iso-estragol), and probably asarone.

Among the allyl derivatives found in natural essences are: chavicol, estragol, eugenol, methyl-eugenol, ethyl-eugenol, betelphenol, safrol, and apiol. C. A. M.

The Volumetric Estimation of Aniline. M. François. (*Journ. Pharm. Chim.*, 1899, ix., 521-524.)—The following method is based on the action of bromine on aniline, as in the equation

$$C_6H_5NH_2 + 6Br = 3HBr + C_6H_2Br_3NH_2.$$

An aqueous solution of bromine is standardized, preferably with a solution of pure aniline hydrochloride prepared by crystallizing the salt two or three times and drying it over sulphuric acid. A solution of aniline hydrochloride containing 1·392 grammes per litre corresponds to one of 1 gramme of aniline in a litre.

In making an estimation, 10 c.c. of the solution of aniline hydrochloride are placed in a flask, and after the addition of two drops of a solution of indigo sulphate the solution of bromine is run in until the indigo is decolorized. The titration is then repeated with another 10 c.c., but this time the bromine-water is run in rapidly to within a few tenths of a c.c. of the first reading; the indigo sulphate is then added, and the titration completed.

An aliquot portion of the solution of aniline under examination, containing, according to a preliminary test, about 0·01 gramme, is titrated in precisely the same manner as in standardizing the bromine, and the percentage calculated.

The author has proved that the volume of the water in which the aniline is dissolved has no influence on the results, and has obtained the same figures with 0·01 gramme of aniline in 5 c.c. or 50 c.c.

When the aniline solution contains mineral compounds, a direct determination is not possible by this method. In such cases the aniline should be liberated by potassium hydroxide, distilled in a current of steam, and estimated in the distillate.

C. A. M.

A Colour Reaction distinguishing Metaphenylenediamine Hydrochloride from Paraphenylenediamine Hydrochloride. L. Cuniasse. (*Ann. de Chim. Anal.*, 1899, iv., 156, 157.)—The reagent consists of a 1 per cent. solution of acetaldehyde in 50 per cent. alcohol, with the addition of a few drops of acetic acid.

On adding to an aqueous solution of metaphenylenediamine hydrochloride several drops of this reagent, warming the mixture, and allowing it to cool, a brilliant yellow coloration with a marked green fluorescence is obtained.

Under the same conditions the hydrochloride of paraphenylenediamine gives a reddish-orange colour, without any trace of fluorescence. C. A. M.

Determination of the Iodine Value of Fatty Acids. A. Zega and R. Majstorovic. (*Chem. Zeit.*, 1899, xxiii., 597.)—The authors record some experiments in which they determined the iodine value of the fatty acids derived from (chiefly) maize-oil on samples of the acids that had been recovered several days previously. These gave figures 10 to 20 points below the normal, the deficiency rising with the time and temperature to which the acids had been exposed. It is highly important, therefore, that this estimation shall always be made under strictly uniform conditions [or immediately the acids are extracted ?]. F. H. L.

On the Baudouin Reaction. **W. Kerp.** (*Zeit. für Untersuch. der Nahr. und Genussmittel*, ii., 473-480.)—The work described in this paper was undertaken primarily with the object of assisting in the study of the question as to whether (as has been advanced on the strength of the Baudouin reaction), on feeding cows upon sesame, the active constituent of the oil passes into the milk. The author has studied the conditions under which furfural produces a red colour with hydrochloric acid, and the relation which this coloration bears to the sesame-oil reaction.

Commercial furfural is usually yellow or yellowish-brown, and requires to be twice distilled under reduced pressure in order to obtain a perfectly pure product. It is a very unstable body. On standing, it gradually acquires a yellow colour and an acid reaction, but the change can be retarded either by keeping the bottles in which it is stored as full as possible, or by the addition of a proportion of alcohol. Twenty per cent. solutions stored in full bottles in the dark keep exceedingly well. Solutions of 1 c.c. of furfural in 100 c.c. of alcohol remain almost completely unchanged, even when not kept exclusively in the dark. Furfural dissolves easily in strong hydrochloric acid. The solution, however, quickly acquires a violet colour, changing to red, and finally to black, with the separation of a black precipitate, and the same changes take place when hydrochloric acid is added to alcoholic solutions of furfural. The reaction depends upon various conditions, such as the absolute quantity and the degree of dilution of the furfural, and the quantity of hydrochloric acid used. It differs, however, from the sesame-oil reaction in the length of time which elapses before the coloration makes its appearance, the sesame-oil reaction being much quicker, and altogether more sensitive. Temperature has also considerable influence upon the course of the reaction. The experiments made in this direction show the necessity of carrying out the sesame-oil reaction at as near the ordinary temperature as possible. Experiments on the action of hydrochloric acid of different strengths on the same alcoholic furfural solution of 0·99 per cent. (volume) showed that acid of 1·127 sp. gr. produced no red-violet colour, even at increased temperature; only a blackish-gray colour developed after standing for eighteen hours, or on warming for half an hour. A comparison of the effects of different strengths of acid showed that while the Baudouin reaction would take place with acid of 1·127 sp. gr., it was less decided than when stronger acid was used, and quickly changed. In doubtful cases it is recommended to use acid of 1·16 sp. gr.

The smallest quantity of sesame oil which can be detected with certainty at the ordinary temperature lies between 0·005 and 0·0025 gramme, provided that the concentration of the sesame-oil mixture does not fall below 2·5 per cent. In proportion as the sesame-oil mixture is more dilute than this, the absolute quantity of the sesame oil must be increased, in order to obtain distinct coloration. In practice, 2 to 2½ grammes of sesame oil, corresponding with 20 to 25 grammes of margarine containing 10 per cent. of sesame oil, can be detected with certainty in 1 kilogramme of butter.

There seems to be no particular advantage in Sohn's proposal to use furfuramide in place of furfural. It is neither so easy to prepare in a pure state as furfural, nor does it keep better. H. H. B. S.

The Action of Silver Nitrate on the Fatty Acids of Cotton-seed Oil. E. Charabot and March. (*Bull. Soc. Chim.*, 1899, xxi., 552-554.)—In 1895 J. Dupont (*Bull. Soc. Chim.*, xiii., 696 and 775) discovered a volatile sulphur compound in cotton-seed oil, but did not determine its nature. The authors have therefore made experiments on the subject with a view of throwing light on the reaction which takes place in Milliau's modification of Becchi's test.

The fatty acids from 50 grammes of the oil were treated on the water-bath with a 30 per cent. solution of silver nitrate, and the resulting brown precipitate washed with hot water and alcohol, and extracted successively with petroleum spirit, benzene (free from thiophene) and ether. The residue was a homogeneous dark-brown substance, consisting principally of the silver salt of a fatty acid and of silver sulphide. This fatty acid melted at 52° C., and solidified at 52° C. It formed a white ammonium salt, which on treatment with silver nitrate gave a brown precipitate resembling the original brown residue.

As Dupont and Charabot have found in olive oil a volatile sulphur compound analogous to that of cotton-seed oil, the authors point out the necessity of caution in accepting the results of the silver nitrate test for the detection of cotton-seed oil in olive oil. C. A. M.

The Determination of the Molecular Weight of Soaps in Alcoholic Solution. F. Krafft. (*Berichte*, 1899, xxxii., 1584-1596.)—It is well known that aqueous solutions of soap undergo decomposition more readily than alcoholic solutions, and from a number of conclusions based on experiment the author has arrived at the theory that when a soap is dissolved in water it is dissociated into its separate components, the molecules of which revolve round each other in small enclosed paths forming part of a system.

This decomposition does not take place in alcoholic solution to any appreciable extent, and it is therefore possible to determine the molecular weight of soaps in such solution by the elevation of the boiling-point in the usual manner.

In a former communication (*Berichte*, xxix., 1328), a number of determinations were made by this method with sodium oleate dissolved in dilute alcohol, and the conclusion was arrived at that its molecular weight was double that calculated from the accepted formula. The author now finds that if absolutely anhydrous alcohol and dry soap be used the molecular weight is in close agreement with the calculated value, and that therefore molecular weights calculated from solutions which contain water have no real significance, since the soap is present in the solution in an incomplete colloidal condition.

The following results were obtained by the ebullioscopic method with potassium acetate (molecular weight, 98·18) dissolved in anhydrous absolute alcohol.

Alcohol. Grammes.	Potassium Acetate. Grammes.	Elevation of Boiling-point, °C.	Calculated Molecular Weight.
19·04	0·3586	0·232	93·3
19·04	0·7229	0·460	94·9
19·04	1·1755	0·734	96·7

The molecular weights found in this way with other pure soaps examined were: Potassium heptylate, $C_7H_{13}O_2Na$ (168·28), found 153·7 to 156·5; sodium laurate,

$C_{12}H_{23}O_2Na$ (222·3), found 237·2; sodium myristate, $C_{14}H_{27}O_2Na$ (250·3), found 253; sodium palmitate, $C_{16}H_{31}O_2Na$ (278·4), found 282·6; sodium oleate, $C_{18}H_{33}O_2Na$ (304·4), found 301·3 to 345·9; potassium oleate, $C_{18}H_{33}O_2K$ (320), found 347.

The author also shows in this paper that the salts of higher fatty acids do not raise the boiling-point of water when dissolved in it, and that the molecular weight of a crystalloid salt such as sodium chloride or bromide can be determined by the ebullioscopic method when dissolved in a not too concentrated soap solution. If, however, the soap be present in sufficient quantity to cause the solution to gelatinize or be salted out by the crystalloid salt added, the steam gives up so much heat to the membrane that the rise in temperature produced by the crystalloid is lost. This was found to be especially the case with potassium stearate, and it was necessary to use only moderately concentrated solutions of this in order to obtain the correct boiling-point on the addition of potassium bromide. C. A. M.

Estimation of Fat in Glue. W. Fahrion. (*Chem. Zeit.*, 1899, xxiii., 452.)—Ten grammes of the sample are broken up and stirred on the water-bath with 40 c.c. of 8 per cent. alcoholic soda till the alcohol has volatilized. If solution is not complete, more spirit is added and the evaporation repeated. The residue is taken up in hot water, and any insoluble matter which is inorganic in character is dissolved in hydrochloric acid. The whole is heated nearly to the boiling point for half an hour, rinsed into a separating funnel, and, when cold, extracted with ether and allowed to rest over-night. The two liquids are run off, the insoluble oxy-fatty-acids in the funnel dissolved in warm alcohol, mixed with the ethereal solution, evaporated, weighed, ignited, and weighed again. The acids, less the ash, give the fat in the glue; but two errors appear—the glycerin of the glycerides is not determined, and the oxy-fatty-acids are slightly soluble in acid. The deficiency, however, is very small, and the process gives higher results than that of Kissling. F. H. L.

Estimation of Indican. K. A. Wolowski. (*Wratsch*, 1899, xx., 482; through *Chem. Zeit. Rep.*, 1899, 170.)—Ninety c.c. of average urine, as passed throughout the twenty-four hours (diluted if dark in colour with 45 c.c. of water), are mixed with 10 c.c. of a 25 per cent. solution of lead acetate. The liquid is filtered, and divided in test-tubes into quantities of 5 c.c. each. To three tubes are added 0·05, 0·1, and 0·15 c.c. of a solution of potassium hypochlorite containing 1 per cent. of active chlorine, and the mixtures are allowed to rest for two or three minutes. Five c.c. of hydrochloric acid (specific gravity 1·19) are introduced and agitated with the samples; after four or five minutes 1 c.c. of chloroform is run in, and the tubes are inverted ten times, but not shaken. If the amount of indican is normal—6 or 7 milligrammes per twenty-four hours—the first tube exhibits a faint colour, the second a stronger one, and the third none at all. To obtain more accurate results, the chlorine reagent may be diluted to 0·1 per cent. in strength. For the calculation of the indican, only its proportion in reference to the solid matter in the urine is taken into account, the latter being deduced by Haisser's factor 2·33. Thus, if the specific

gravity of the urine is 1·018, it contains (18 × 2·33 =) 44 grammes per litre. In northern climates the mean amount of solid matter is 65 grammes and 0·0065 gramme of indican per litre, which corresponds with 0·76 gramme of chlorine. F. H. L.

The Stability of Oxalic Acid Solutions. W. P. Jorissen. (*Zeits. angew. Chem.*, 1899, 521.)—Although the literature of this subject is somewhat voluminous, the results appear inconclusive and contradictory, and the varying effects of light, moulds, and preservative agents upon standardized oxalic acid of different strengths have not been completely elucidated. For his own experiments, the author has prepared pure oxalic acid by re-crystallization, rejecting the first and last 10 per cent. of the product. The solutions were kept for fifty to one hundred days in bottles, which had been cleaned with boiling water in order to sterilize them and to dissolve superficial alkali, and they were finally titrated with permanganate or with standard soda. His investigations prove that normal oxalic acid is not permanent in light. In the dark, and in the absence of ferments, a solution remains unchanged even when only centinormal in strength. Fifty c.c. of sulphuric acid per litre is an effectual preservative in the dark against moulds and against decrease in strength. One gramme of boric acid is useless; 2 grammes only act efficiently in cold weather. A decinormal solution in 12·4 or 25 per cent. alcohol loses strength in thirty-six days even in the dark. In daylight, sulphuric acid and boric acid both increase the speed of oxidation; manganese sulphate acts similarly in darkness and in light.

F. H. L.

Estimation of Oxalic Acid in Urine. E. Salkowski. (*Centralbl. med. Wissensch.*, 1899, xxxvii., 257; through *Chem. Zeit. Rep.*, 1899, 189.)—Two hundred to two hundred and fifty c.c. of urine, or if the liquid is weak 500 c.c., are evaporated to one-third their bulk, mixed with 20 c.c. of hydrochloric acid (specific gravity 1·12), and extracted with 20 to 25 c.c. of ether saturated with alcohol, three such extractions being made, in order to separate the oxalic acid from the insoluble phosphoric acid. The united extracts are filtered through a dry paper and freed from solvent; the residue is diluted with water and evaporated to about 20 c.c., then filtered to remove resinous substances. The filtrate is made slightly alkaline with ammonia, treated with 1 or 2 c.c. of a 10 per cent. solution of calcium chloride, rendered acid with acetic acid and the calcium oxalate collected and weighed. F. H. L.

Detection and Estimation of Bile Pigments in Urine. A. Jolles. (*Zeits. physiol. Chem.*, 1899, xxvii., 83; through *Chem. Zeit. Rep.*, 1899, 148.)—For the detection of bile pigments, about 10 c.c. of urine are shaken with 1 c.c. of chloroform and 4 or 5 c.c. of 10 per cent. barium chloride solution. After standing a few minutes to settle, the liquid is pipetted off, and the residue agitated with 2 or 3 c.c. of Hübl's centinormal iodine solution and 1 c.c. of strong hydrochloric acid. In presence of bile pigments the precipitate, the chloroform solution, and the liquid floating on the latter are all coloured green or greenish-blue; traces of pigments cause the precipitate alone to be greenish.

For the quantitative estimation of bilirubin, 10 or 20 c.c. of urine are measured into a pear-shaped separating funnel with a very short tube at the base, mixed with 20 c.c. of chloroform, 10 c.c. of 10 per cent. barium chloride, and 50 c.c. of 1 : 5 hydrochloric acid. After agitation and separation 15 c.c. of the chloroform are run off into a graduated tube, and the few drops remaining in the stem of the funnel are returned by inverting the vessel and opening the stopcock; another 15 c.c. of chloroform are added, shaken up, and 12 c.c. drawn off; 10 c.c. of chloroform are then added, and 8 c.c. finally drawn off. The united extracts are washed with 30 c.c. of 1 : 1 hydrochloric acid to remove traces of urine mingled with the precipitate in the chloroform, and the solution is next brought into a stoppered flask by the aid of two rinsings with 25 c.c. of alcohol. 10 c.c. of centinormal Hübl solution are agitated with the liquid for five minutes, then 5 c.c. of 10 per cent. potassium iodide, 5 c.c. of fresh starch and 100 c.c. of water are added, and the whole is titrated with centinormal thiosulphate till the red-brown liquid above the chloroform is completely decolorized. Towards the end of the process this is best ascertained by allowing the solid matter to settle, withdrawing two samples of 2 or 3 c.c. of the clear liquid, dropping a little thiosulphate into one, and noting any further disappearance of colour. F. H. L.

[Thudichum has denied (*J. pr. Chem.*, 1896, [2], liii., 314) that bilirubin can be quantitatively oxidized to biliverdin by iodine in alcoholic solution (*cf. J. Chem. Soc. Abs.*, 1894, ii., 466, and 1896, i., 516).—ABS.]

Determination of Phenols in Urine. C. Neuberg. (*Zeits. physiol. Chem.*, 1899, xxvii., 123; through *Chem. Zeit. Rep.*, 1899, 189.)—The ordinary process of Kessler and Penny for the estimation of phenols in urine is incorrect when the liquid contains grape-sugar, so that the increased secretion of phenols which has been noted in cases of diabetes may be an analytical error. The following method avoids the defect. As in the Kessler and Penny process, the urine is freed from acetone, the phenols are liberated from their sulphonic acids, distilled off, and rectified over calcium carbonate to remove nitrous and formic acids. They are next brought into a 2-litre flask and mixed either with 3 grammes of air-dried lead hydroxide (freshly precipitated from the nitrate with baryta-water) and 5 c.c. of a strong solution of basic lead acetate, or with a solution of 1 gramme of sodium hydroxide and 6 grammes of lead acetate. The whole is heated on a boiling water-bath to yield lead phenolates and to volatilize aldehydes, the latter being completely removed by boiling for about five minutes over a naked flame until a few drops of the distillate no longer reduce ammoniacal silver solution. The contents of the flask are then rendered strongly acid with sulphuric acid, and the phenols are distilled over, repeating the operation twice again after water has been added to restore the original volume. This distillate is examined by the Kessler and Penny process; *i.e.*, it is made alkaline, warmed on the water-bath, treated with excess of decinormal iodine, cooled, and titrated with decinormal thiosulphate. Useful results can also be obtained without distillation by removing the lead sulphate, making the filtrate alkaline, and titrating as before; the titration cannot, however, be conducted in the presence of the sulphate. F. H. L.

INORGANIC ANALYSIS.

Detection and Estimation of Mercury Vapour in Air. Kunkel and Fessel. (*Verhandl. physik. med. Gessell. Würzburg*, 1899, xxxiii., 1; through *Chem. Zeit. Rep.*, 1899, 189.)—Fifty or a hundred litres of air dried over calcium chloride are led at a speed of eight or ten litres per minute through a dry tube 2 or 3 millimetres in diameter containing a few grains of iodine. Even 0·01 milligramme of mercury will be indicated by the formation of the red mercuric iodide; or, if the speed of passage be raised considerably and the quantity of iodine increased, by a partial production of the yellow iodide. To determine the proportion of mercury, the solid matter from the tube is taken up in a little potassium iodide solution, the undissolved iodine filtered off, the dissolved iodine combined with sodium hydroxide, the liquid treated with sulphuretted hydrogen, and the colour matched by means of a standard solution of mercuric chloride handled in a similar manner. One cubic metre of air will retain 6·7 to 14 milligrammes of mercury at temperatures of 16° to 23° C. when saturated therewith. Gold-leaf also removes mercury vapour completely.

F. H. L.

Estimation of Total Phosphoric Acid in Thomas Meal. C. Aschmann (*Chem. Zeit.*, 1899, xxiii., 435.)—To avoid the unpleasantness of working with strong hot acids, the following method of bringing Thomas meal into solution is advocated. A liquid containing 420 grammes of pure nitric acid (sp. gr. 1·2) and 50 grammes of concentrated sulphuric acid per 10 litres is prepared. Five grammes of the sample are agitated with 500 c.c. of the mixture for half an hour in Wagner's apparatus, at a speed of forty to forty-five revolutions per minute. The mass is filtered immediately, allowed to rest for twelve hours for the calcium sulphate to settle; then 50 c.c. are filtered off, treated with 10 c.c. of a solution of 500 grammes of citric acid in 1 litre of water, made just alkaline with ammonia (using a fragment of litmus-paper as indicator), and diluted with 50 c.c. of water. If the liquid remains perfectly clear for twenty minutes, the phosphoric acid is precipitated with Wagner's magnesia mixture, stirred for half an hour, and filtered after one or two hours' standing (longer is not harmful); but if silica falls during the twenty minutes, the nitro-sulphuric acid solution must first be precipitated with Wagner's molybdate. The said solution becomes cloudy after a time, owing to the deposition of free sulphur produced by the action of the nitric acid on the nascent sulphuretted hydrogen, but this is of no importance. Nitro-sulphuric acid dissolves some 90 per cent. of Thomas meal, the liquids filter easily, and the results agree well with the ordinary methods.

F. H. L.

Influence of Bacteria on the Decay of Cement. (1) **A. Stutzer and R. Hartleb.** (*Zeits. angew. Chem.*, 1899, 402); (2) **G. Barth.** (*loc. cit.*, p. 489.)—In 1896 Stutzer observed that the cement mortar which had been in use for eight or nine years as lining to a water-supply reservoir was converted into a brownish mud containing less lime and more iron oxide, etc., than the original cement. This disintegration he ascribed, and still believes to be primarily due, to the solvent action of the carbon dioxide in the water, which dissolved the lime of the cement in the form of bicar-

bonate. But on extracting further samples of the mud from the same reservoir lately with sterilized water, the present authors found that it was highly charged with nitrifying organisms; so much so, that when introduced into a 0·1 per cent. solution of ammonium sulphate, the ammonia reaction practically disappeared in fourteen days and nitrite reactions were given powerfully. A similar experiment with asparagine only gave a nitrite reaction after six weeks, but here it was necessary for ammonia first to be formed before nitrification could set in. A further test with a 0·1 per cent. solution of sodium nitrite showed that nitrates were formed but slowly. It would appear possible, therefore, in the case of waters containing any appreciable proportion of oxidizible nitrogenous matter, that this production of nitrous acid by the agency of micro-organisms may play some part in the destruction of cement. The parallel action that is noticed in sea-water, unless the cement is fortified by the addition of extra active silica, is due rather to the soluble sulphates than to carbon dioxide, which is not present in such large quantities as in fresh water; it has not been feasible to determine whether bacteria assist in this disintegration likewise.

With reference to this matter, Barth quotes another instance of cement decomposition occurring in three years where the water contained no unusual amount of carbon dioxide, and he seems to consider the previous explanation plausible. His figures for the original cement and for the soft brown mud are as follows:

	Cement.	Mud.
SiO_2	20·0%	33·21%
Al_2O_3	7·5%	21·84%
Fe_2O_3	3·5%	10·95%
CaO	63·0%	24·23%
MgO	2·0%	9·36%

F. H. L.

APPARATUS.

Bruhns' Improved Polarimeter. (*Zeits. angew. Chem.*, 1899, 694.)—In this apparatus the scale is engraved or etched on the long travelling quartz wedge itself, so that want of coincidence between the wedge and the scale is no longer possible. By this arrangement one single calibration of the scale suffices for the tabulation of its errors, and each time the apparatus is required for use it is only necessary to bring the zero-point on the short fixed wedge into unison with the zero-point of the travelling scale. In front of the polarimeter a disc moving on a pin eccentric to the axis of vision carries the telescope and a magnifier, either of which can be brought into the central position by turning a handle. The magnifier is focussed on the scale, while the telescope, being focussed on the far nicol, renders the scale temporarily invisible, and prevents it being in the way during adjustment. But as in half-shadow apparatus the scale is masked in the final position, the aforesaid moving disc is connected through its spindle with another quartz plate, which is brought into the field between the analyser and the wedge simultaneously with the magnifier, thus lighting the scale again without affecting its readings. The whole of the optical part of the apparatus is protected by a padded case to prevent changes of temperature and the frame of the wedge also carries a thermometer. F. H. L.

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OCTOBER, 1899.

THE ANALYSIS OF GOLDEN SYRUP.

By R. Bodmer, Norman Leonard, and H. M. Smith.

As the analysis of "golden syrup" has recently called for considerable attention on the part of public analysts, we venture to think that an account of the methods employed and the results obtained in the laboratory of Dr. Thomas Stevenson and in that of one of us (R. B.) may be of service to other members of the Society.

It would seem from the convictions which have been obtained under the Sale of Food and Drugs Act that "golden syrup" is required to be the product of the partial hydrolysis of cane-sugar (sucrose), and should therefore consist only of cane- and invert-sugar, besides water, mineral matter, and small quantities of organic impurities. A large proportion of the syrups now sold are, however, either mixtures of cane-sugar syrup with "glucose syrup" or else consist merely of "glucose syrup" sweetened by the addition of cane-sugar. These products are characterised by the presence of dextrin, maltose, and excess of dextrose. Since these last-named compounds are dextro-rotatory, dextrin being so to a very high degree, a determination of the rotatory power of the sample will at once afford a clue to its nature. Assuming genuine golden syrup to have a specific rotation $[\alpha]_D = +16°$ and glucose syrup $[\alpha]_D = +110°$, the percentage of glucose syrup present will be:

$$\frac{100([\alpha]_D - 16)}{110 - 16}.$$

A more extended examination is, however, usually desirable, and the following method of analysis has been found to be convenient.

Water.—A 10 per cent. solution of the syrup is made, and from the specific gravity (water = 1000) of this solution the solid matter, and hence the water, in the original syrup is calculated by using the well-known factor 3·86.

Rotatory Power.—The 10 per cent. solution is clarified, if necessary, by means of a little animal charcoal, placed in the tube of the polarimeter, and the angle of

rotation (a) observed. The specific rotation of the syrup is then calculated by the well-known formula,

$$[a]_D = \frac{100a}{lc},$$

where l is the length of the observation tube in decimetres and c is the concentration (grammes per 100 c.c.). Using a 2 decimetre tube and a 10 per cent. solution, the observed angle has simply to be multiplied by 5.

Reducing Power.—This is determined (a) in the original solution after suitable dilution, (b) after inversion of the cane-sugar, (c) after inversion of the cane-sugar, dextrin, and maltose.

(a) Ten c.c. of the 10 per cent. solution are diluted to 100 c.c., and the reducing power determined by means of Fehling's solution. We prefer the gravimetric process, using 20 c.c. of Fehling's solution for 10 c.c. of the diluted sugar solution.

(b) Ten c.c. of the 10 per cent. solution are diluted to 50 c.c. with water containing 5 c.c. of concentrated hydrochloric acid, heated for ten minutes at a temperature not exceeding 68°, cooled, rendered alkaline, and made up to 100 c.c. Ten c.c. of this solution may be treated with 25 c.c. of Fehling's solution.

(c) Ten c.c. of the 10 per cent. solution are diluted to 50 c.c. with water containing 1½ c.c. of concentrated sulphuric acid, heated on the water-bath for two to three hours, cooled, rendered alkaline, and diluted to 100 c.c. Thirty c.c. of Fehling's solution should be used for 10 c.c. of this liquid.

The reducing powers thus determined are expressed as percentages of glucose in the original syrup, and denoted by the symbols K_1, K_2, and K_3 respectively.

Ash.—Ten c.c. or 20 c.c. of the solution of the syrup are evaporated on the water-bath with a drop or two of concentrated sulphuric acid, and the residue ignited and weighed. The usual deduction of one-tenth is to be made from this "sulphated" ash.

Dextrin.—In the presence of dextrin, a turbidity is produced on adding alcohol to the solution of the syrup, but no great importance is to be attached to this test, since a similar turbidity has been noticed in the case of syrups made from coarse sugar, where there was no other evidence of the presence of dextrin. The quantitative estimation of dextrin by precipitation with alcohol was, as might be expected, found unreliable. The fermentation method is probably more accurate, but we have had no personal experience of this.

In the following table are given the results obtained by the application of the methods described to a number of samples received for analysis under the Sale of Food and Drugs Act. All were sold as "golden syrup" with the exception of No. 2, which was labelled as "amber syrup."

No.	Water.	Ash.	Specific Rotation. $[\alpha]_D$.	Reducing Power. Original. K_1.	After Inversion of Cane-sugar. K_2.	After Prolonged Hydrolysis. K_3.
1 ...	16·0	2·0	+15°	45·7	80·8	
2 ...	20·5	1·2	+86°	39·5	49·2	79·9
3 ...	16·9	1·4	+68°	39·4	52·9	73·0
4 ...	17·9	0·5	+105°	33·2	38·5	80·3
5 ...	20·6	0·5	+62·5°	38·2	60·7	81·5
6 ...	19·4	1·0	+100°	36·8	42·0	78·1
7 ...	20·4	0·5	+67·5°	34·4	64·0	76·5
8 ...	17·6	1·8	+82·5°	37·7	50·7	74·7
9 ...	18·5	2·4	+92·5°	30·2	44·9	79·7
10 ...	21·9	1·1	+100°	35·5	38·8	78·7
11 ...	19·6	1·5	+99°	34·2	41·8	78·1
12 ...	13·8	8·2	+17·5°	32·5	66·6	72·8
13 ...	16·2	4·1	+11·5°	45·6	72·8	70·8
14 ...	16·3	2·1	+13°	45·3	81·2	77·4
15 ...	19·7	1·8	+95·5°	35·7	43·1	82·4
16 ...	20·1	7·9	+25°	22·0	64·9	57·2
17 ...	23·7	0·6	+90°	39·5	42·2	74·4
18 ...	19·8	1·5	+94·5°	32·2	40·6	74·4
19 ...	19·8	1·6	+82°	29·1	46·8	75·6
20 ...	20·5	1·8	+91°	31·7	36·7	71·2
21 ...	16·5	2·0	+12·5°	44·0	73·9	
22 ...	16·9	1·8	+16°	40·8	77·5	78·0
23 ...	19·8	1·5	+90°	29·0	41·7	65·3
24 ...	19·6	1·4	+100°	34·0	39·0	70·3
25 ...	15·4	3·4	+57°	35·8	56·2	78·9
26 ...	16·0	3·5	+75°	32·9	48·8	75·1

Of these samples, Nos. 1, 12, 13, 14, 16, 21 and 22 are, in all probability, unadulterated syrups prepared from more or less refined sucrose. Assuming the original reducing power to be due to invert-sugar and the increase in the reducing power on inversion to be due to cane-sugar, the composition of the genuine syrups may be expressed as in the following table:

	1.	12.	13.	14.	16.	21.	22.
Water	16·0	13·8	16·2	16·3	20·1	16·5	16·9
Ash	2·0	8·2	4·1	2·1	7·9	2·0	1·8
Cane-sugar	33·3	32·4	25·8	34·1	40·8	28·4	34·9
Invert-sugar	45·7	32·5	45·6	45·3	22·0	44·0	40·8
Organic matter other than sugar (by difference)	3·0	3·1	8·3	2·2	9·2	9·1	5·6
	100·0	100·0	100·0	100·0	100·0	100·0	100·0
Specific rotation, calculated ...	11·6°	14°	6·6	12·2°	22·1°	8·7°	13·8°
,, ,, observed ...	15°	17·5°	11·5	13°	25°	12·5°	16°

It will be noted that the specific rotation calculated on the above assumption is always lower than that actually observed, the difference being probably caused by substances included under the head of "organic matter." No. 13 was shown to be a beet-sugar product by the development of a slight fishy odour in the solution on keeping, by the high ash, and by the very small amount of phosphoric acid present (0·009 per cent. P_2O_5).

All the remaining samples are, to a greater or less extent, adulterated with glucose syrup, as is shown by the high rotatory power and the increase in reducing power after prolonged hydrolysis. The original reducing power (K_1) is here due, not only to invert-sugar, but also to dextrose (from starch) and maltose, whilst the increase in reducing power on prolonged heating with acid is caused by the inversion of dextrin and maltose. It should be theoretically possible to calculate the amounts of all these substances from the values obtained for the reducing power and the specific rotation of the mixture before and after the two inversions described above, but up to the present we have found that the errors of experiment and the disturbing influence of impurities are too great to allow of the necessary number of equations being obtained with sufficient accuracy for the desired purpose. Failing this, however, a convenient expression of the composition of the sample may be arrived at by treating anhydrous commercial "glucose" as an individual substance having a cupric reducing power of 53 and a specific rotation +134° for sodium light. (See L. K. Boseley, ANALYST, 1898, p. 125.)

If s is the percentage of cane-sugar (K = O, $[\alpha]_D = +66{\cdot}5°$), i that of invert-sugar (K = 100, $[\alpha]_D = -23{\cdot}1°$), x the commercial glucose, then the following relations hold:

$$K_1 = i + 0{\cdot}53x.$$

$$K_2 = i + 0{\cdot}53x + \frac{100s}{95}.$$

$$[\alpha]_D = \frac{66{\cdot}5s + 134x - 23{\cdot}1i}{100}.$$

Whence:

$$s = \frac{95(K_2 - K_1)}{100}.$$

$$x = 0{\cdot}685[\alpha]_D + 0{\cdot}16K_1 - 0{\cdot}455s.$$

$$i = K_1 - 0{\cdot}53x.$$

The following table shows the results obtained by the application of these formulæ to a few typical samples:

	5.	3.	2.	15.	24.	4.
Water	20·6	16·9	20·5	19·7	19·6	17·9
Ash	0·5	1·4	1·2	1·8	1·4	0·5
Cane-sugar	21·4	12·8	9·2	7·0	4·8	5·0
Invert-sugar	17·3	14·4	7·2			
"Glucose"	39·4	47·1	61·0	67·9	71·8	75·0
Organic matter (by difference)	0·8	7·4	0·9	3·6	2·4	1·6
	100·0	100·0	100·0	100·0	100·0	100·0

Since cane-sugar syrup contains from one-third to one-half its weight of cane-sugar, and glucose syrup contains an average of 82 per cent. of solid matter, these samples may be represented as containing approximately :

	5.	3.	2.	15.	24.	4.
Cane-sugar syrup ...	55	40	25	20	15	10
Glucose syrup	45	60	75	80	85	90

ON THE DETERMINATION OF THE IODINE VALUE.

By J. Lewkowitsch.

(*Read at the Meeting, June* 7, 1899.)

It is not my intention to add a new modification to those that have been published since the appearance of Hübl's classical paper on the determination of the iodine values of fats and oils; but as the theory of Hübl's process has at last reached a satisfactory solution, chiefly through the papers published by Wijs, I thought it might be useful to summarize the present position of this question.

The papers of Wijs have been fully abstracted in this journal, and I need therefore only refer to them (Analyst, 1898, xxiii., 238, 240). They corroborate Ephraim's experiments (Analyst, 1895, xx., 176) that ICl is added on to the unsaturated carbon atoms. It is true that Wijs assumes that the active agent in the Hübl solution is IOH; yet, as this leads to a somewhat artificial explanation of the chemistry of the process, inasmuch as some postulates demanded thereby are not borne out by experiment, it will be sufficient for practical purposes, as leading to the same analytical result, to adopt the explanation that ICl is absorbed by each pair of doubly-linked carbon atoms. Wijs proposed at first to use a solution of ICl in 95 per cent. acetic acid, but he has more recently replaced this by a solution of ICl in glacial acetic acid, as he must have found, like myself, that the former solution does not possess the stability he claimed for it. This is proved by the following figures, obtained in my laboratory by Dr. Peck :

Effect of Time on Wijs' Iodine Solution.

Time.	Solution of $I_2 + Cl_2$ in 95 per cent. Acetic Acid.	Solution of $I_2 + Cl_2$ in 99 per cent. Acetic Acid.	Solution of $ICl_3 + I_2$ in 99 per cent. Acetic Acid.
	Grammes of I, 1 c.c. of the solution is equal to :		
Original value ..	0·02682	0·02550	0·0259
After 16 hours ...	0·02642	—	—
After 40 hours ...	0·02625	—	—
After 64 hours ...	0·02581	—	—
After 2 months ...	—	0·02554	0·0258
After 3 months ...	0·01997	—	—
After 5 months ..	0·01911	—	—

The solution of ICl in glacial acetic acid is infinitely superior to the original Hübl solution as regards stability; furthermore, as it can be prepared rapidly, and as the time in testing is very much shortened, the process must be considered a very valuable one, all the more so as Wijs' maintains that his iodine values are probably the most nearly correct. As I have been using the Hübl solution for a number of years, and have determined the iodine constants for a great number of fats and oils by means of this method, it seemed to me necessary to ascertain whether all these iodine numbers could still be regarded as correct, especially so as Wijs had shown in one of his publications (ANALYST, 1898, xxiii., 241) that the iodine numbers obtained by his method for ten different oils were invariably higher by several units than those recorded by Hübl's process. Special stress has been laid on the fact that Wijs obtained for a sample of allyl alcohol the iodine value of 436·8 after ten minutes, whereas the Hübl solution gave him 425 after twenty hours' standing, whilst I had published a few years ago the iodine values of 349 to 376.* Since I calculated from some experiments given in Wijs' first paper the iodine value of 392, and, furthermore, since determinations made with different samples of allyl alcohol have no decisive value, I considered it necessary to examine, conjointly with Wijs, one and the same sample, although it is easy to see that allyl alcohol is not a specially suitable substance to decide the questions at issue.

Wijs was good enough to send me a sample of allyl alcohol which gave him by his method the iodine number 429. The experiments carried out in my laboratory by Dr. Peck gave the following results:

IODINE VALUES OF ALLYL ALCOHOL.

WIJS' METHOD.		HÜBL METHOD.
Solution $ICl_3 + I_2$.	Solution $I_2 + Cl_2$.	Solution two to three days' old.
419·09 419·09	420·09	421·27 421·27 421·87 422·17

I add some experiments also made by Dr. Peck on pure elaïdic acid, M.P., 44·5° C.:

IODINE VALUE OF ELAÏDIC ACID.

WIJS' METHOD.		HÜBL METHOD.
Solution $ICl_3 + I_2$.	Solution $I_2 + Cl_2$.	Solution two to three days' old.
88·66	88·96	88·24
88·93	88·89	—
—	89·98†	89·01†
—	89·98†	89·01†

These experiments, as also a further number of determinations made with cotton-seed stearin, prove conclusively that iodine determinations made with Hübl solution lead to correct results, provided that the Hübl method is applied in a proper

* This sample cannot have been pure, it must have contained some moisture.

† In these experiments the substance was made up to a known volume in the solvent, and aliquot parts of this were used in the test.

manner. This method has been described fully by me elsewhere, and I need not therefore refer to it here at any length. My conclusion that both methods yield practically identical results has since been fully corroborated by Wijs himself, who has shown the reason why different results were obtained by different observers when working with Hübl solution, although individual observers working under exactly the same conditions obtained identical results. He has also proved experimentally that the time during which the Hübl solution is allowed to act must not be chosen arbitrarily, and that it makes a considerable difference whether the blank test is done at the commencement or at the finish of the actual test; and he has further shown that the Hübl solution must have been allowed to stand one day before use, in order to attain a certain degree of stability, if I may so term it, and that this solution should not be allowed to act any longer than six or seven hours on the fat under examination. If these rules are adhered to, both solutions yield identical results.

In conclusion, I wish to tender my best thanks to Dr. E. L. Peck for his valuable assistance in the preparation of this paper.

DISCUSSION.

The CHAIRMAN (Mr. ALLEN) said that he had met with glacial acetic acid which contained a considerable proportion of sulphurous acid. It was purified almost entirely by partially freezing it, and allowing the unfrozen portion to drain off. Obviously the presence of such an impurity in the acetic acid would very greatly reduce the figure obtained in the original iodine titration.

Dr. LEWKOWITSCH said that, as has been done already by Wijs, great stress must be laid upon the importance of testing the glacial acetic acid, and ascertaining that it had no reducing effect upon permanganate.

ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

FOODS AND DRUGS ANALYSIS.

Volumetric Estimation of Alcohols, especially of Fusel Oil in Brandy. F. Adam. (*Oesterr. Chem. Zeit.*, 1899, ii., 241.)—This process depends on the fact that acetyl chloride dissolved in chloroform reacts with alcohols, forming neutral esters (acetates); so that 1 molecule of the chloride mixed with 1 molecule of a monovalent alcohol liberates 1 molecule of hydrochloric acid (equivalent to 1 molecule of alkali), while 1 molecule of acetyl chloride mixed with 1 molecule of water yields 1 molecule of acetic acid and 1 molecule of hydrochloric acid (equivalent to 2 molecules of alkali). A 250 c.c. flask is joined by a well-fitting cork to a 100 c.c. separating-funnel, and both are thoroughly dried. The alcoholic liquid is placed in the flask, 25 c.c. of a 5 per cent. solution of acetyl chloride in chloroform are added, the funnel is put in position with its cock closed and half filled with water. After standing two hours, a set of Varrentrapp's bulbs charged with water (to retain any

escaping hydrochloric acid vapours) is connected with the top of the funnel, the stopcock is cautiously opened, and the water is allowed to descend into the flask. When the excess of chloride is decomposed, the mixture of chloroform and water with the water from the bulbs is titrated with normal alkali and phenolphthalein. In the meantime, the same volume of acetyl chloride solution (25 c.c.) is treated with 100 c.c. of water and titrated similarly, the difference between the two tests multiplied by the molecular weight of the alcohol giving the amount of the latter. Blank experiments on pure amylic alcohol show fairly satisfactory results.

To separate the fusel oil from the ethylic alcohol in brandy, the sample is diluted till its strength is about 20 per cent. by volume. It is shaken rapidly with exactly 25 c.c. of specially purified chloroform, the solvent run off, and a second 25 c.c. employed as before. The 50 c.c. of extract are agitated three times with 100 c.c. (each) of water, allowed to settle thoroughly, finally dried with anhydrous calcium sulphate, and filtered. Thirty-five c.c. of the chloroform are then examined as above. The chloroform should be purified by warming over acetyl chloride, followed by washing with alkaline water, and distillation over calcium chloride. An allowance of 0·3 c.c. of normal alkali must be made for the above quantities of brandy, etc., even in the absence of fusel oil, and this figure is to be deducted from the difference between the two titrations. The examples show a maximum error of 0·0072 gramme (mean 0·0031) in estimating from 0·03 to 0·3 gramme of amylic alcohol.

In an experiment in which 0·1992 gramme of amylic alcohol and 0·133 gramme of essential oil of caraway had been added to 200 c.c. of brandy, after distillation over potassium hydroxide, 0·198 of amylic alcohol was found. Evidently the addition of an essential oil of this class does not interfere with the determination, its phenol-like constituents and the acids combined therewith, are retained by the alkali. In the case of the esters of the higher alcohols of the fatty series, obviously the reverse would hold; but if the distillation over alkali were omitted, then these esters would exercise no disturbing action.

Experiments with absolute ethylic alcohol give errors ranging from −0·0086 to +0·0176 gramme in dealing with 0·228 to 0·346 gramme. Phenol cannot be determined in this manner; but other alcohols and mixtures thereof, or mixtures of alcohols with other bodies, should be amenable to the treatment, provided they are soluble in chloroform. In such cases the quantity of acetic acid neutralized may be expressed as a percentage of the alcohol or ethereal oil, and termed the "alcohol number."

The acetyl chloride solution should not be used till it has been kept one or two days, in order to give the water, alcohol, and hydrochloric acid opportunity to deposit or escape; after that it alters less in strength than might be imagined. F. H. L.

Detection of Liquorice in Wine. G. Morpurgo. (*Oesterr. Chem. Zeit.*, 1899, ii., 361.)—When a normal wine is treated with magnesia in the cold no ammonia is given off, but if the sample has been mixed with liquorice, at least in such proportion as will increase appreciably the amount of extract, ammonia is evolved distinctly. Two grammes of magnesium hydroxide and 25 c.c. of wine are placed in a 150-c.c. flask

closed with a cork, from which is suspended a moist strip of red litmus-paper. If the litmus remains unchanged for ten minutes liquorice is practically absent. Several samples of Malaga evolved ammonia, but these were of unknown origin; three specimens from a firm of good repute yielded none. Some samples of Turkish wine with a high content of extract also gave a blue coloration in half an hour; but all the other varieties of wine examined by the author liberated no ammonia, whereas the addition of 0·50 gramme of liquorice per litre determined a reaction in two minutes.

In doubtful cases, or where greater certainty is required, the glycyrrhizin must be tested for. 100 to 250 c.c. (according to the proportion of extractive matter) of the wine are mixed with powdered glass, evaporated, preferably on the water-bath, to a syrup, and strongly acidified with citric acid; the mass is rubbed down with alcohol, the solution filtered, and the residue extracted twice again. The filtrates are concentrated to a syrup and treated with 20 c.c. of alcohol and 50 c.c. of ether. After standing some time, the presence of liquorice is demonstrated by a brown deposit or skin which adheres strongly to the vessel. The liquid is poured off, the flask dried on the water-bath, and the mass dissolved in a little water. On adding ammonia it becomes greenish-brown, and after evaporation exhibits the characteristic taste and odour. A wine rich in extract and colouring matter may also leave a similar residue, but in this case it tastes harsh, and not sweet. If the aqueous solution is diluted with more water before introducing the ammonia, it becomes turbid if much liquorice is present; further dilution and gentle warming make the glycyrrhizin to cohere into a black resin-like mass, while the wine substances remain dissolved. To exclude the presence of dulcin or saccharin, the ethereal liquid poured away from the residue is evaporated, the solid matter is dissolved in water, mixed with a few drops of basic lead acetate, and evaporated again. When taken up in alcohol and the solution filtered, a colourless filtrate should result, evaporating without appreciable residue. If a residue be left, it should be examined for dulcin and saccharin according to the usual methods. F. H. L.

The Composition and Analysis of Chicory. J. Wolff. (*Ann. de Chim. Anal.*, 1899, iv., 157-162 and 187-193.)—Commercial chicory is the roasted product of the root of *Cichorium intybus*, which in the cultivated state the author found to weigh in the mean 325 grammes, whilst the root of wild-chicory from the outskirts of Paris weighed scarcely 30 grammes.

Starch does not appear to be present in chicory, but there is a considerable proportion of inulin $[6(C_6H_{10}O_5)+H_2O]$, the percentage being almost identical in the cultivated and wild products.

In the author's opinion it is probable that the small quantities of reducing sugar noted by other observers in chicory consist of lævulose derived from the inulin, and he considers it doubtful whether saccharose is a constituent.

According to Lescœur and Morelle, the specific rotatory power of inulin is $[\alpha]_D = -36·57°$.

It can be obtained from the root by extracting it with hot water containing a little calcium carbonate to prevent a partial conversion into lævulose, concentrating

the filtered liquid, and precipitating the inulin with alcohol. A small quantity of other substances is simultaneously extracted, including soluble proteids and a slight amount of synanthrose or lævulin, which has no action on polarized light, and which does not reduce Fehling's solution. On boiling with dilute acids synanthrose is converted into dextrose and lævulose.

In the analysis of green chicory 2·16 grammes of the freshly-pulped substance were mixed with 150 c.c. of water and a little calcium carbonate, and the liquid heated for two hours on the water-bath at 80° to 90° C., and finally brought to the boiling-point and left to cool.

After the addition of 10 c.c. of a concentrated solution of basic lead acetate and an excess of a concentrated solution of sodium sulphate, the volume of the liquid was made up to 200 c.c., and filtered. The reducing sugar was then determined by titration with Fehling's solution and by the polarimeter. By heating 50 c.c. of the solution with 5 c.c. of hydrochloric acid at 69° to 70° for twenty minutes, the inulin was completely converted into lævulose, which was determined polarimetrically and by titration with Fehling's solution. The difference between the reducing sugar found before and after inversion gave the lævulose corresponding to the inulin (180 of lævulose = 162 of inulin).

The method employed by the author to determine the effect of roasting on the chicory was similar to that described above, and is best shown by an example. Seven grammes of the finely-divided substance (18 per cent. of water) gave a solution containing 17·1 per cent. of reducing substances before decolorization and 10·9 after decolorization, the difference (6·2) being taken as caramels. The additional amount of reducing substances obtained after inversion corresponded to 7·2 per cent. of inulin.

When inulin is heated at gradually increasing temperatures, it produces lævo-rotatory products, which gradually give place to dextro-rotatory substances. When gently warmed it yields a small quantity of a gummy lævo-rotatory substance—pyrinulin. The effect of prolonged heating is to produce a very large proportion of pyrinulin and of dextrins (which do not reduce Fehling's solution) at the expense of the reducing substances.

In the subjoined table of the results of the analysis of different varieties of commercial chicory, the soluble matter was determined by extracting 2 grammes of the sample with a litre of boiling water, the extraction being repeated with more water until the extract was colourless. The residue was dried in the oven on a filter of known weight, and weighed, and from the result the soluble matter plus moisture was calculated.

The water was estimated by exposing a weighed quantity of the crushed substance in a vacuum over sulphuric acid for four or five days, and finally drying it at 100° to 110° for twelve hours.

The cellulose was determined directly on the substance by a method not mentioned.

For the estimation of the iron 10 grammes of the material were incinerated, the ash taken up with warm hydrochloric acid, and the solution diluted and filtered. A large excess of hydrochloric acid was added to the filtrate, and the iron titrated in the boiling liquid by means of a solution of stannous chloride standardized on copper.

Analysis of Chicory.

	Moi	Ash.	Sodium Chloride.	Iron.	Lævulose nd Dextro	Inulin.	Caramels	Aqu... Extract.	Cellulose.	Salt	Total Nitrogen.	Soluble Nitrogen.	Extrs Mat
Green chicory (cultivated) ...	79·2	1·11	—	—	0·60	13·15	—	—	1·29	0·11	1·15	—	17·12
The same dried	17·0	—	—	—	5·3	47·51	—	—	—	—	—	—	—
The same roasted	16·0	2·75	—	—	14·4	9·6	9·0	61·0	9·1	1·7	6·15	3·2	64·2
Green chicory (wild)	80·4	—	—	—	1·1	12·4	—	—	—	--	—	—	—
Commercial chicory ...	13·3	5·9	0·22	0·15	12·4	4·3	11·6	59·3	6·9	1·7	5·5	2·5	66·4
,, ,, ...	9·2	6·3	0·30	0·07	7·5	5·0	14·7	54·3	13·2	—	6·0	2·4	63·0
,, ,, ...	14·0	4·6	0·22	0·05	14·2	9·6	12·8	65·9	6·5	2·6	6·6	4·0	65·5
,, ,, ...	14·5	3·7	0·18	0·03	9·0	4·0	15·6	61·3	11·1	2·7	6·1	2·8	60·7
,, ,, ...	10·7	8·5	0·17	0·29	12·4	6·6	12·3	59·8	8·5	2·3	6·3	3·6	63·5

C. A. M.

On the Active Principle of Cayenne Pepper. K. Micko. (*Zeit. für Untersuch. der Nahr. und Genussmittel*, ii., 411.)—In a former paper (*Zeit. für Untersuch. der Nahr. und Genussmittel*, i., 818), the author published the results of an inquiry into the active principle of *Capsicum annuum*. He now shows that the same body—capsaicin ($C_{18}H_{28}NO_3$)—is also the active principle of *Capsicum fastigiatum*. The melting-point of capsaicin from *Capsicum annuum* was found to be 63·5° C., that from *Capsicum fastigiatum* 64° C. The reactions of the two bodies were identical; both gave the characteristic vanilla-like odour on dissolving in alcohol, decomposing with platinum chloride and evaporating, and both showed the same crystalline form, coefficients of solubility and chemical composition. H. H. B. S.

Determination of Digestible Nitrogenous Matter and Albuminoid Nitrogen in Foods and Feeding Stuffs. B. Sjollema. (*Zeit. für Untersuch. der Nahr. und Genussmittel*, ii., 413-417.)—Stutzer's method for the determination of digestible albumin by the use of gastric juice has certain drawbacks. (1) The preparation of the fluid is unpleasant. (2) A fluid of sufficiently active properties cannot always be obtained, unless the stomachs of several pigs are used. (3) There is a difficulty in preparing fluid of uniform activity—a necessary requirement in making a long series of experiments. (4) Whenever a fresh fluid is prepared, it is necessary to determine the nitrogen contained therein, in order that the necessary correction may be made. These disadvantages may be overcome by substituting dry pepsin for Stutzer's fluid. The procedure recommended is as follows: Two grammes of the substance under examination are digested with 430 c.c. of water, 1 gramme of pepsin, and 16 c.c. of 10 per cent. hydrochloric acid, for forty-eight hours, at a temperature of 38° to 40° C., with frequent shaking. Three times during the digestion 11 c.c. of 10 per cent.

hydrochloric acid are added—viz., at the expiration of sixteen, twenty-four, and forty hours respectively from the commencement. The fluid is then made up to 500 c.c. The nitrogen is determined by Kjeldahl's method both in the insoluble residue as well as in the fluid. Comparative results by the two methods are given in the following table:

Description.	Nitrogenous Matter Dissolved on Digesting with		Difference.	Nitrogenous Matter left Undissolved on Digesting with		Difference.
	Gastric Fluid. Per cent.	Artificial Pepsin. Per cent.	Per cent.	Gastric Fluid. Per cent.	Artificial Pepsin. Per cent.	Per cent.
Wheat ...	10·20	10·20	0	0·60	1·00	+0·40
Horse-beans...	23·85	24·15	+0·30	2·15	1·90	−0·25
Dried carrots	4·70	5·25	+0·55	0·95	1·10	+0·15
Earthnut cake	47·85	48·05	+0·20	1·65	1·75	+0·10
Cotton-seed meal	47·30	47·55	+0·25	3·85	3·55	−0·30
Linseed cake, I.	40·00*	40·20*	+0·20	2·30	2·30	0
,, ,, II.	36·00*	35·40*	−0·60	2·35	2·45	+0·10
,, ,, III.	34·90*	35·50*	+0·60	2·30	2·20	−0·10
Dried potatoes	6·35	6·25	−0·10	0·85	1·10	+0·25

A modification of Stutzer's method for the determination of pure albumin is also proposed. In dealing with such substances as linseed cake, there is a difficulty in applying the copper method, owing to the slimy nature of the fluid obstructing the separation of the precipitate. The difficulty can be overcome by proceeding as follows: One gramme of substance is boiled with 50 c.c. of water, and, while boiling, 50 c.c. of 95 per cent. alcohol are gradually added. Fifty c.c. of cold water, 2 drops of a cold-saturated solution of alum, and the necessary quantity of copper solution are then added, after which the process is continued in the usual way. The filtration is carried out by means of a porcelain funnel, with the help of a filter-pump. In the examples given, the results agree well with those obtained by Stutzer's original method. H. H. B. S.

The Determination of Morphine in Opium. C. Montemartini and D. Trasciatti. (*Gazzetta Chim. Ital.*, 1899, xxix., 292-300.)—In a former communication (*Gazzetta Chim. Ital.*, xxvii., 302-335) the authors described a process of estimating morphine, which they termed the "sodium chloride method." In the present paper they reply to the criticisms of H. Thoms and of K. Dieterich (*Ber. d. deut. Pharm. Ges.*, viii., 124, 171), and point out that their directions were inaccurately translated into the German. A number of experiments are described to show that concordant results can readily be obtained, and finally the following outline of their process is given, including the modifications which their recent work has suggested.

Ten grammes of the finely-powdered opium are dried at 100° C., and macerated at intervals for an hour with 90 to 100 c.c. of a 20 per cent. solution of sodium

* Calculated upon the dry substance.

chloride. The supernatant liquid is filtered off, and the mass drained on the filter and again digested with about 60 c.c. of the same solution of sodium chloride. This digestion and filtration are repeated until the filtrate no longer gives any reaction with Fröhde's reagent.

The combined extracts are evaporated to dryness on the water-bath and the residue dried in the oven, finely pulverized, and extracted with boiling absolute alcohol until morphine is no longer removed. The alcohol is evaporated from this extract and the residue mixed with 15 c.c. of water rendered slightly alkaline with ammonium hydroxide, so that the odour of ammonia is still perceptible after the liquid has stood for twenty-four hours in an exposed place with occasional agitation.

The precipitate is collected on a weighed filter, washed, until the washings are colourless, with cold water previously saturated with morphine, dried at 100° C., and weighed. It is then transferred to a stoppered funnel and washed with successive portions of chloroform or benzene until, on evaporating a few drops of the filtrate and adding hydrochloric acid and then sodium hydroxide, no turbidity results. The filter is then again dried at 100° C. and weighed. C. A. M.

Estimation of Morphine in Opium. H. M. Gordin and A. B. Prescott. (*Arch. Pharm.*, 1899, ccxxxvii., 380; through *Chem. Zeit. Rep.*, 1899, 234.)—Three grammes of opium are rubbed down with a few c.c. of a mixture composed of 5 vols. of strong ammonia, 5 vols. of alcohol, 10 vols. of chloroform, and 20 vols. of ether. More of the liquid is then added till the opium is flooded, the vessel is covered up, and put on one side for five or six hours. 10 or 20 grammes of powdered sodium chloride are next stirred in, and the basin is placed in a current of air (stirring at intervals) till the solvents have evaporated. The residue is put in a desiccator overnight, the lumps are broken up, and the powder is brought into a percolator, rinsing out the vessel with common salt. The mass is then extracted very slowly with benzene so long as 10 drops of the liquid after evaporation and acidification yield a turbidity with 2 or 3 drops of Wagner's reagent. When all the opium bases excepting the morphine are thus shown to be dissolved, the solid matter is extracted with a mixture of 5 vols. of chloroform and 1 of alcohol until all the morphine is dissolved (using Wagner's test again). The solution is collected in a flat basin, the solvents are volatilised in a current of air, the residue is rubbed up with 50 c.c. of $\frac{N}{20}$ sulphuric acid, and brought into a graduated cylinder with the aid of water, where it is finally diluted to 90 c.c. 75 c.c. of this liquid (equal to 2·5 grammes of opium) are mixed with 35 c.c. of $\frac{N}{20}$ potassium hydroxide, and the excess of alkali is titrated with equivalent acid, using methyl orange paper as indicator. The volume of $\frac{N}{20}$ acid absorbed by the alkaloid is calculated into morphine by multiplying by

$$(0{\cdot}0142 \times \frac{100}{2{\cdot}5}) = 0{\cdot}568.$$

F. H. L.

Detection and Estimation of Ergot in Meal. F. Musset. (*Pharm. C. H.*, 1899, xl., 353; through *Chem. Zeit. Rep.*, 1899, 190.)—A mixture of chloroform and alcohol (about 10 : 1) is prepared having a specific gravity 1·435. Five grammes

of the sample are shaken with 60 c.c. of the liquid, and the whole is allowed to settle in a vertical tube; the meal sinks, the ergot and a few scales float. The floating particles and 2 or 3 c.c. of the solution are brought into another tube, mixed with the similar product from a second 5 grammes, and treated with alcohol till everything sinks. The liquid is poured away, the deposit placed on a microscope slide, and examined in xylene with a power of 800 diameters. The sclerotia appear as dark or almost black bodies; and when examined by reflected light they appear yellow with a tinge of green, thus being distinguishable from other opaque substances.

With the object of estimating the proportion of ergot, the author determines the cornutine, and calculates it according to the ratio 0·2 : 100. An amount of meal corresponding to 200 grammes of the dry material is spread out in a basin and placed for two hours under a bell jar, which also covers a small vessel containing ammonia. It is next shaken for an hour with 200 c.c. of ether, placed in a percolator, and treated with the same solvent till 450 c.c. have passed through. The ether is repeatedly extracted with 30 c.c. of 0·5 per cent. hydrochloric acid; the aqueous liquid is made alkaline with ammonia and extracted with ether, this treatment with ether being repeated several times. The final ethereal liquids are divided into two portions. The residue of one is moistened with 5 drops of strong sulphuric acid, and stirred with a rod moistened with ferric chloride, which gives a visible reaction with 0·1 milligramme of cornutine. The other half is extracted with hydrochloric acid; the ether dissolved by the liquid is evaporated, the aqueous solution diluted to 50 c.c., and mixed with 3 drops of potassium mercuric iodide. If only a faint turbidity is produced, the meal contains less than 0·1 per cent. of ergot. A strong turbidity is matched colorimetrically by tests with 50 c.c. of 0·5 per cent. hydrochloric acid and 3 drops of the double iodide containing from 0·3 to 0·5 milligramme of cornutine. F. H. L.

TOXICOLOGICAL ANALYSIS.

Notes on the Guaiacum Reaction for the Detection of Blood. E. Schaer. (*Amer. Journ. Pharm.*, 1899, lxxi., 361-370.)—From the author's experiments the following appears to be the most simple and reliable method of applying this test for the detection of blood in stains: The coloured spots are first moistened with acetic acid, and then extracted either with a 70 per cent. chloral hydrate solution, or directly with a 1 per cent. solution of guaiacum in chloral hydrate (70-75 per cent.). In the former case, which the author regards as preferable, an equal volume of the guaiacum chloral solution is added to the extract. This process has the advantage that if the stain happened to contain nitrites, the nitrous acid liberated by the acetic acid would give an immediate blue coloration with the guaiaconic acid of the guaiacum. Moreover, if the chloral hydrate extract contain only blood, the addition of the guaiacum solution to it will give a pale brown mixture, well adapted for obtaining a decisive zone reaction indicating the presence of hematin. This is tested for by pouring a layer of Hünefeld's turpentine solution, or of a solution of hydrogen peroxide, which has been proved to give no reaction with guaiacum, on the surface of the mixed liquid, when an intense blue will be formed at the junction of the liquids.

A suitable Hünefeld's solution may be prepared by mixing 15 c.c. of turpentine oil which has been exposed for some time to the light and air, and which does not change directly to blue in the guaiacum tincture, or 15 c.c. of a 3 to 5 per cent. solution of hydrogen peroxide, free from acids, with 25 c.c. of alcohol, 5 c.c. of chloroform, and 1·5 c.c. of glacial acetic acid.

In cases of very small stains, it is advisable to digest them in a flat porcelain dish with strong chloral hydrate solution, after first moistening them with acetic acid, and after thirty minutes' contact to pour on to the digested mass a correspondingly small quantity of the chloral hydrate solution of guaiacum, and when thoroughly mixed to add a few drops of the oxidizing reagent.

The author has found that very old blood-stains may be identified in this way, provided that a sensitive solution of the guaiacum prepared from quite fresh resin be used, and that the Hünefeld's solution, or solution of hydrogen peroxide, do not themselves give any coloration with guaiacum.

The zone coloration as thus obtained is much more stable than that obtained in the older methods, owing to the fact that its formation is progressive.

With regard to the possibility of confusing the blood reaction with the blue coloration given by the numerous compounds which directly colour the guaiacum resin, such as free halogens, permanganic acid, ferric and cupric salts, quinone, etc., the author remarks that in their presence the guaiacum tincture would *at once* become blue *before the addition* of Hünefeld's solution. In the case of other substances, however, such as white blood cells, and pus cells, and animal and vegetable enzymes, a false interpretation of the guaiacum blue reaction might be made. But the author has found that the "oxygen-transferring" power of the enzymes is destroyed or greatly weakened at 100° C., or by contact with dilute hydrocyanic acid. So that if the stain contained substances of the nature of enzymes instead of the constituents of blood, it will cease to give the guaiacum reaction after a short digestion on the hot-water bath, and a control experiment with hydrocyanic acid will give essentially negative results.

It is stated in conclusion that guaiaconic acid, as employed by Doebner (*Archiv. d. Pharm.*, 1897), gives a specially pure blue coloration when dissolved in 200 to 500 parts of chloral hydrate, and when the reaction is observed in a test-tube as a zone reaction. C. A. M.

ORGANIC ANALYSIS.

A Colour Reaction for Vinyl Alcohol. **E. Rimini.** (*Gazzetta Chim. Ital.*, 1899, xxix., 390-93.)—In the course of an examination of various methods of estimating fat in milk and cheese, the author obtained a violet coloration in Bondzynski's method, which is based on the Werner-Schmidt process (*cf.* Seyler, ANALYST, xx., 156).

This coloration was readily obtained by shaking a small quantity of cheese in a test-tube with hydrochloric acid (specific gravity 1·19) so as to dissolve the casein, adding a few drops of ether, again shaking and gently heating.

Having found that with pure ether recently distilled with lime no reaction was obtained, the author made a number of experiments with substances which might

possibly be present as impurities, such as methyl, ethyl, and amyl alcohols, aldehyde, furfural, formic, acetic, sulphurous, and ethyl sulphuric acids, and acetone, but in each case the results were negative.

Poleck and Thümmel have found that commercial ether invariably contains traces of hydrogen peroxide and of vinyl alcohol ($CH_2=CH.OH$), and the latter substance was found to be the cause of the reaction. It can be detected by adding mercury oxychloride to the ether, which gives a white precipitate of mercury vinyl oxychloride By distilling the ether over alkali this impurity is eliminated, and no coloration is given by the distillate in the Werner-Schmidt method.

Two theories are suggested to explain the formation of vinyl alcohol in ether. According to Schaer, it is due to the slow oxidation of the ether by the air, as in the equation

$$(CH_3 \cdot CH_2)_2 : O + O_3 = (CH_2 : CH \cdot OH)_2 + (OH)_2,$$

while, according to Richardson, the atmospheric oxygen, assisted by light, acts upon the moisture in the ether, forming hydrogen peroxide, and this in turn may oxidize the ether with the formation of vinyl alcohol.

The substance in the cheese taking part in the reaction was attributed to the casein or its decomposition products. Peptones were found to give a marked coloration, but if the decomposition of the proteid molecule had proceeded as far as the formation of amido-compounds or of phenols there was no reaction.

In the author's opinion these observations throw light upon Liebermann's reaction, according to which certain proteid bodies, when washed with alcohol and cold ether and heated with concentrated hydrochloric acid (1·19 specific gravity), give a violet coloration. As he obtained Liebermann's coloration when ordinary commercial ether was used, but not with purified ether, he considers that there can be no doubt that the reaction is due to the presence of traces of vinyl alcohol in the ether. C. A. M.

A Reaction for Ketones. Gillet and Hairs. (*Bull. de l'Ass. belge*, 1899, xiii., 170.)—On adding a large excess of Nessler's reagent to an aqueous solution of dimethylketone, a lemon-yellow crystalline precipitate is formed after a few moments. This reaction is also given by all the other ketones examined by the authors. It is extremely sensitive, detecting the presence of one drop of acetone in a litre of water, but in the presence of a large proportion of acetone the precipitate is not formed.

The compounds formed appear to be analogous to those obtained with Denigés' mercuric reagent (ANALYST, this vol., p. 93). C. A. M.

A Colour Reaction of the Benzoyl Compounds. G. Denigés. (*Bull. Soc. Pharm. Bordeaux*, March, 1899; through *Ann. de Chim. anal.*, 1899, iv., 199, 200.)—The reagent (1 c.c. of 30 per cent. formaldehyde in 50 c.c. of sulphuric acid) used by the author for the differentiation of different polyphenols (*Bull. Soc. Pharm. Bordeaux*, 1898, 241) can also be used for the detection of the benzoyl radicle (C_6H_5CO) in organic compounds. On warming a trace of the substance with 2 or 3 c.c. of this reagent in a tube containing a thermometer, a reddish-brown colour appears at about

120° C., and the liquid shows a large but not well-defined absorption-band in the green part of the spectrum.

This coloration is readily given by benzoyl chloride, benzoic acid, benzoyl acetic anhydride, benzamide, hippuric acid, and cocaine.

The same colour is produced by other compounds, but at temperatures other than 120° C. Thus, phenol, polyphenols and the opium alkaloids react in the cold, whilst salicylic compounds are coloured below 100° C.

The presence of the benzoyl group may be masked if the other groups of the compound give the reaction below 120° C., as is the case, for example, with benzaldehyde. C. A. M.

A New Method of Estimating Hydrazine. E. Rimini. (*Gazzetta Chim. Ital.*, 1899, xxix., 265-269.)—This is based on the reduction of iodic acid by the hydrazine and the titration of the unreduced acid with thiosulphate after elimination of the free iodine.

The reaction takes place in accordance with the equation

$$3N_2H_4 + 2HIO_3 = 3N + 6H_2O + 2HI.$$

The solution of the hydrazine salt is mixed with an excess of a solution of potassium iodate of known strength, and the liquid heated over a direct flame until the whole of the liberated iodine is expelled and the solution becomes colourless. It is then cooled, acidified with sulphuric acid, and titrated with decinormal thiosulphate.

It is stated that bromates are also reduced by hydrazine, but only on heating, while chlorates are not attacked.

The author has observed similar reactions in the case of hydroxylamine, phenylhydrazine, and semicarbazide, and is studying the applicability of his method to the determination of those compounds. C. A. M.

The Determination of the Dropping-point of Ceresin and Paraffin. Finkener. (*Mittheilungen aus. d. Königl. Techn. Versuchsanstalt*, 1899, vii., 100-103.)—On warming a mixture of chemically pure substances of different melting-points which have no chemical action upon one another except solvent action, the temperature remains almost constant about the melting-point of the lower melting constituent until this has ceased to dissolve the more solid constituent, and then rapidly rises.

As it is difficult in such cases to determine the exact melting-point, the author considers that the best method of determining the temperature at which the substance changes its state of aggregation is by taking its dropping-point under constant conditions.

He has therefore laid down the following conditions for the guidance of the Customs authorities in determining the dropping-point of ceresin and paraffin: The dropping-point is the temperature at which a drop of the substance under examination falls from the end of a glass rod 3 millimetres in diameter when slowly heated in a test-tube 250 millimetres long and 30 millimetres wide. The test-tube is immersed to four-fifths of its depth in water, the temperature of which is raised

about 1° C. per minute, and the thermometer is placed beside the rod in such a manner that its bulb is the same distance (about 30 millimetres) from the bottom of the tube.

In order to insure uniformity in the size of the drops, the substance is melted on the water-bath and the glass rod dipped into it to a depth of 10 millimetres, so that on solidification the adherent drop forms a hemisphere on the end of the rod.

In conclusion the author gives tables of the results of parallel determination of the dropping-point of a sample of ceresin, which tend to show that slight variations in the conditions, such as having the test-tube 26 millimetres wide instead of 30 millimetres, and having the thermometer in different positions in the tube, do not cause any considerable deviation.

A variation in the rate at which the temperature of the water is raised slightly lowers the dropping-point, as is shown in the following results :

Per Minute.	0·5° C.	1° C.	1·5° C.	2° C.
	75·1	75·0	74·6	74·1
	74·7	74·8	74·8	74·5
	75·0	75·1	74·8	74·3
	74·8	74·7	74·6	74·3
Mean ...	74·9	74·9	74·6	74·3

The author states that his results were confirmed with other samples of ceresin and paraffin. C. A. M.

On the New Method of Examining Ceresin used by the German Customs. **D. Holde.** (*Mittheilungen aus. d. Königl. Techn. Versuchsanstalt*, 1899, vii., 103-108.) —In order to distinguish ceresin from mixtures of ceresin and paraffin, the Custom House officials are directed to take the dropping-point of the sample in question (see preceding abstract). If this be 66° C., or higher, they may conclude that the substance is ceresin derived from ozokerite; whilst if it be lower than 66° C., the sample must be regarded as a mixture of ceresin and paraffin.

In criticising these directions the author points out that mistakes may readily be made by relying on the dropping-point as a conclusive test. Thus, mixtures of paraffin and ceresin can be prepared with dropping-points considerably higher than 66° C. Moreover, mixtures of pure ceresin and carnauba wax may "drop" far above that temperature, and the presence of paraffin in the mixture has but little influence in counteracting the effect of the carnauba wax. Then there are pure paraffins which melt above 66° C., as, for instance, some from Boghead coal, which in the author's experiments did not melt below 80° C., and others from Halle which had dropping-points of 65·7° C. and 66·4° C. Finally, it is possible to prepare from crude ozokerite of poor quality pure specimens of ceresin which themselves melt far below 66° C. These points are illustrated in a series of tabular results.

As regards the method of determining the dropping-point, the author remarks that a number of precautions are necessary if concordant results are to be obtained. Thus, if too much ceresin be attached to the end of the rod, the substance will drop too soon. On the other hand, if there be too little, the substance may be heated far

above its melting-point without falling. In order to avoid the necessity of making a large number of determinations, he recommends in preparing the drop, dipping the rod twice in the melted substance, and allowing the adhering material to cool to the temperature of the room before the second dipping.

It is further shown that the shape of the bulb of the thermometer and the distance of the mercury from the sides of the tube have more or less influence on the results, and it is therefore advisable to invariably use a thermometer with a cylindrical bulb about 5 millimetres in diameter, and to fix both the glass rod and the thermometer at a distance of about 10 millimetres from the sides of the tube by means of a cork support above them. C. A. M.

The Detection and Estimation of Traces of Iodine in Organic Substances. P. Bourcet. (*Bull. Soc. Chim.*, 1899, xxi., 554-556.)—The finely divided substance is fused with potassium hydroxide in a nickel basin. The fused mass is extracted with boiling water, and the solution thus obtained concentrated to half its volume. Dilute sulphuric acid (1 : 5) is then added to the cold liquid, care being taken to avoid heating. When neutral, the solution is again rendered alkaline with potassium hydroxide, and gradually mixed with half its volume of 95 per cent. alcohol.

The greater part of the potassium sulphate is precipitated, and is filtered off and washed with 30 per cent. alcohol. The filtrate is evaporated to a third of its original volume, and a fresh quantity of 90 per cent. alcohol added to it when cold. This precipitates a further amount of potassium sulphate, which is removed and washed as before. By repeating the concentration of the filtrates and precipitation with alcohol several times, the iodine is obtained in the alkaline liquids free from all potassium sulphate.

The filtrates are evaporated to dryness, and the residue slightly ignited, to destroy any residual organic matter. The residue is taken up in as small a quantity of hot water as possible, filtered, and a few c.c. tested for iodine by means of nitrous acid and carbon disulphide.

A table is given showing the amount of iodine per kilogramme determined by this method in various kinds of edible fish. The results vary from 0·2 milligramme in the fresh ray to 1·9 milligramme in the mussel and 2·0 milligrammes in the herring. C. A. M.

A. E. Shuttleworth's Method of determining Ash in Organic Substances B. Tollens. (*J. Landwirthsch.*, 1899, xlvii., 173; through *Chem. Zeit. Rep.*, 1899, 205.)—When vegetable or animal bodies are ignited, there is always a liability that the temperature may rise too high, and the ash sinter or fuse. In this case some of the silica may combine with the bases to form silicates insoluble in hydrochloric acid, so that when the ash is attacked with acid part may be left undissolved, and the percentage of "silica" appear higher than it should. To prevent this danger, Shuttleworth treats 5 or 6 grammes of the material to be incinerated with 20 c.c. of a 1 per cent. solution of calcium acetate and a sufficiency of water to moisten it thoroughly, then dries the mixture and ignites it. The calcium acetate not only

renders the mass more refractory, but also hastens the combustion. The reagent, which must obviously be pure, is best prepared from marble and hydrochloric acid, removing the iron, precipitating the calcium as oxalate, washing, igniting in a muffle, and dissolving the residue in dilute acetic acid to the above-mentioned strength.

Shuttleworth has also designed an apparatus for the ignition of organic matter in which loss of chlorides by volatilization is avoided, and which allows the ash of such substances as straw to be determined in less than four hours; but the furnace is not described in the German abstract. F. H. L.

The Determination of Sulphur in Russian Petroleum. G. Filiti. (*Bull. Soc. Chim.*, 1899, xxi., 338-341.)—The method here described is an application of that employed by Langbein for the estimation of sulphur in saccharin, and consists in oxidizing the substance in a calorimetric bomb in the presence of oxygen and a little water (ANALYST, xxi., 264).

The advantages claimed for this method over that of Carius are that it is possible to use a quantity 20 times as great for the estimation, and that the products of the combustion contain not more than traces of nitric acid, while the results are said to be exceedingly accurate.

The process also appears to be suitable for the estimation of phosphorus and halogens in volatile organic compounds. C. A. M.

The Determination of Total Sulphur in Coal. U. Antony and A. Lucchesi. (*Gazzetta Chim. Ital.*, 1899, xxix., 181-184.)—The following modification of Eschka's method (ANALYST, xiii., 17, 192) is recommended as giving accurate results in a much shorter time. One gramme of the finely-powdered coal is intimately mixed in a platinum crucible with an oxidizing mixture, consisting of manganese dioxide, 4 grammes; potassium permanganate, 1 gramme; and sodium carbonate, 2 grammes. A thin layer of the oxidizing mixture is placed on the top, and the crucible heated for thirty minutes, at first gently, and finally till its bottom is of a red heat.

When cold the oxidized mass is transferred to a beaker containing 40 to 50 c.c. of water, the liquid boiled after the addition of nitric acid, filtered, and the sulphuric acid precipitated with barium chloride in the usual manner.

As manganese dioxide often contains sulphur, a blank determination with that to be used is advisable.

In the analyses of four samples of coal by this method, and by Eschka's, the results agreed closely.

Closely concordant results were also given by the two methods in the analysis of a sample of coal, to which had been added a considerable proportion of free sulphur, combined sulphur (pyrites), and organic sulphur (thiourea). C. A. M.

Reactions distinguishing between the Products of Peptic and Pancreatic Digestion. V. Harlay. (*Journ. Pharm. Chim.*, 1899, ix., 468-470.)—The juice of *Russula delica* contains a tyrosinase, which behaves differently with the products of

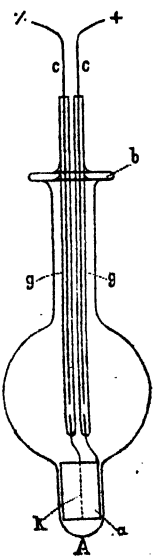
peptic and of pancreatic digestion. With the latter it gives a red coloration, which subsequently becomes black, while with the former it produces a red colour, which changes to green after some time. On treating this green liquid with a few drops of a solution of an alkali it is changed to bright red, the green colour being again restored on acidification. The final brownish-black colour given by pancreatic products does not change on the addition of an alkali.

Another distinguishing reaction is that given by bromine water. On adding to 5 c.c. of the solution containing the products of the pancreatic digestion of albumin or fibrin several drops of a saturated aqueous solution of bromine, there results a reddish-yellow precipitate, which dissolves on shaking, forming a reddish-coloured solution. This reaction is only obtained on adding the bromine little by little, and if too much be added a yellow precipitate is formed, which gradually changes to red and finally to dark violet. The presence of 1 per cent. of hydrochloric acid does not interfere with the reaction.

With the products of peptic digestion, the red coloration is not observed either in the acid solution or in the liquid neutralized with calcium carbonate. In the former case, bromine water does not give any yellow coloration unless it be added in excess, when a yellow precipitate is formed, which remains unaltered for a long time. Added to the neutralized solution, the reagent gives a slight turbidity, with a very faint violet coloration, whilst if it be added in excess, there is a yellow precipitate.

C. A. M.

An Electrolytic Method of Determining Nitrogen in Organic Substances. **C. C. Budde and C. V. Schou.** (*Zeit. anal. Chem.*, 1899, xxxviii., 344-48.)—The application of electrolysis to the estimation of nitrogen has been found by the authors to have considerable advantages over the usual methods of estimation. In their experiments they have made use of the apparatus shown in the accompanying figure. This consists of a round-bottomed flask, the bulb of which is made as small as possible, and, as a rule, contains not more than 50 c.c., although with substances which froth much it may be as large as 100 c.c. The neck of the flask is about 2 centimetres in diameter, and there is an extension in the bottom, A, holding about 10 c.c. The weighed substance is placed in this, and well mixed with 4 c.c. of fuming sulphuric acid and 8 c.c. of the ordinary concentrated acid, after which the electrodes are introduced.



The anode consists of a cylinder of platinum foil, *a* (about 0·1 millimetre thick, 50 millimetres wide, and 30 millimetres long), to the longest side of which is attached a platinum wire about 5 millimetres in length. The cathode, *k*, is a thick platinum wire about 18 millimetres long and 1 millimetre thick, which is fused on to a finer wire and then hammered out flat. Each of the thin platinum wires is fused into the end of one of the glass tubes, *g*, in which are inserted the copper wires, *c*, and the contact is completed by means of mercury.

The tubes are connected preferably by means of one of the glass attachments, *b*, fused on to each of them. When the electrodes are introduced these

attachments rest on the edge of the flask. On completing the circuit and connecting the copper wires with the poles of a battery of 8 volts, the strength of the current rises in a short time to 10 ampères, and the liquid becomes very hot. After about forty-five minutes the liquid is as a rule quite colourless, while in most cases about half the acid has been evaporated or decomposed.

When cool the liquid is distilled with alkali and the ammonia determined in the usual manner.

The following table gives the results of the determination of nitrogen in various classes of organic nitrogenous substances:

Substance.	Electrolytic Method. Nitrogen, per cent.	Theoretical. Nitrogen, per cent.
Strychnine	8·33	8·38
Morphine	4·70	4·62
Caffeine	28·72	28·86
Acetanilide	10·34	10·37
Diphenylamine ...	8·48	8·28
Asparagine	18·65	18·66
Quinolin tartrate ...	4·63	4·25
Egg albumin	12·55	—
Cocoa	3·33	—
Uric acid	32·78	33·33

It is essential to use a mixture of the fuming and ordinary acids, or the results will be too low, as is shown by a series of determinations. The apparatus here described can be obtained from Max Kaehler and Martini, Berlin. C. A. M.

INORGANIC ANALYSIS.

Estimation of Mercury in Urine by Amalgamation. Schumacher and W. L. Jung. (*Arch. experiment. Pathol.*, 1899, xlii., 138; through *Chem. Zeit. Rep.* 1899, 205.)—Jolles' process for estimating mercury in urine does not give exact results because the amalgamation is not quantitative, unless the gold is employed in a very fine state of subdivision. To bring it into this form, fibres of carefully-purified asbestos are well soaked in an almost neutral solution of gold chloride (prepared from the pure metal, and freed from excess of acids), and the salt is reduced by ignition in hydrogen. This gold-asbestos is used as a filter, and removes the whole of the mercury. Mercury precipitated by means of stannous chloride from urine which has been decomposed by treatment with hydrochloric acid and potassium chlorate still retains organic matter; therefore to obtain it in a form fit for amalgamation the whole process must be repeated a second time. F. H. L.

On the Determination of Potassium as Potassium Platinum Chloride. F. Bolm. (*Zeit. anal. Chem.*, 1899, xxxviii., 348-350.)—The following modification of a process used for a long time past in the laboratory of Fresenius is recommended by the author. The solution of platinum chloride used for the precipitation is diluted to such an extent that the precipitate is only formed on evaporating the liquid. The precipitate is collected on a filter previously treated with hot water and

alcohol, and is washed with 96 per cent. alcohol. After drying off the alcohol, the platinum salt is dissolved off the filter in hot water, evaporated in a glass basin on the water-bath, and dried to constant weight at 130° C.

After this the precipitate may be reduced to metallic platinum by Sonstadt's method, which consists in dissolving the residue in a little hot water and decomposing it with mercury on the water-bath until the supernatant liquid is clear. The weight of the platinum, multiplied by the factor 2·494, gives the amount of the original potassium platinum chloride.

The subjoined table gives the results of comparative determinations by these methods and by the more tedious one, in which the precipitate is weighed on the dried filter-paper :

Weighed on the Filter-paper, K_2PtCl_6 grammes.	Weighed in Glass Basin, K_2PtCl_6 grammes.	Weighed as Platinum, K_2PtCl_6 grammes.
0·0940	0·0936	0·0935
0·2851	0·2855	0·2861
0·1279	0·1276	0·1272

C. A. M.

Estimation of Calcium Carbonate in Soil. A. Stutzer and R. Hartleb. (*Zeits. angew. Chem.*, 1899, 448.)—This process has been designed to meet the requirements of agricultural laboratories, and to answer the question whether a particular soil would be improved by dressing with chalk or marl. The ordinary test of noting any effervescence on addition of acid is only trustworthy when sufficient chalk is present to leave no doubt on the point; and the regular methods of determining carbon dioxide by absorption are too tedious and costly for frequent employment. The process depends on the fact that calcium (or magnesium) carbonate is decomposed by boiling with a large excess (10 parts by weight) of an ammonium salt, evolving the quantitative proportion of ammonium carbonate which can be absorbed in standard acid. From 5 to 20 grammes of the sample are boiled with ammonium chloride and 200 c.c. of water for forty-five minutes in a flask of Jena glass, a blank experiment being made simultaneously in a flask of exactly the same material and with the same description of receiver. The examples quoted show a satisfactory agreement with the ordinary methods of analysis.

In the case of (tropical) soils containing much iron, some of which may exist as carbonate, the disturbing influence of this constituent may be eliminated by boiling the earth with water alone for half an hour so as to decompose the ferrous carbonate, then cooling, adding the ammonium chloride, and proceeding as before. Here the new process has an industrial advantage; for the absorption methods determine all the carbon dioxide, including that of the ferrous carbonate, whereas by volatilization as now described only the normal carbonates of calcium and magnesium are returned, which are precisely the substances of interest to the agriculturist. F. H. L.

Estimation of Carbonates in Soil. A. Mayer. (*Landw. Versuchsst.*, 1899, li., 341; through *Chem. Zeit. Rep.*, 1899, 205.)—The author states that a mixture of 1 part of "acetic acid" with 2 parts of water leaves ferrous carbonate practically unattacked; therefore the useful calcium carbonate, as distinguished from the useless

ferrous carbonate, may be estimated by treating the finely-powdered soil with such acid in Scheibler's apparatus, and calculating from the carbon dioxide evolved.

F. H. L.

The Analysis of Silicon. H. Borntträger. (*Zeit. anal. Chem.*, 1899, xxxviii., 350, 351.)—The following method was used by the author in the analysis of specimens of silicon prepared by the electrolytic process. Ten grammes of potassium hydroxide were dissolved in 100 c.c. of water in a silver basin, which was then gently heated on the water-bath while 0·5 gramme of the finely divided silicon was introduced little by little. After diluting with hot water, the liquid was filtered and the residue washed with hot water until the washings were no longer alkaline. It was then dissolved off the filter with hot hydrochloric acid, the solution evaporated to dryness, and the traces of silica separated in the usual manner. The small amounts of iron and aluminium were precipitated by ammonium hydroxide in the filtrate from the silica.

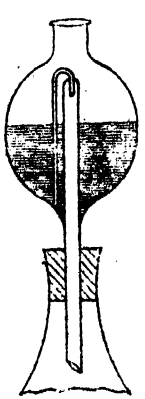
When these were present only in traces they were weighed together as oxides, and calculated into the metal by multiplying the result by 0·6. The difference between the impurities thus determined and 100 was taken as silicon.

In the following representative analysis the iron was determined by reduction with zinc and hydrochloric acid and titration with permanganate: Silicon, 98·53; iron, 1·04; and aluminium, 0·43 per cent.

C. A. M.

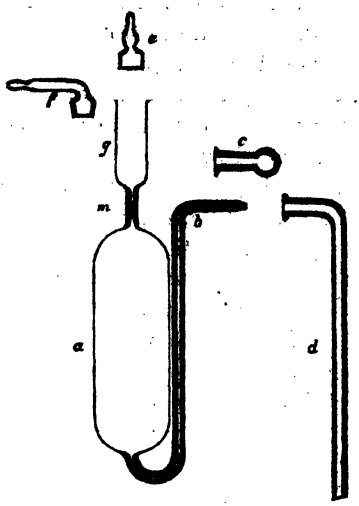
APPARATUS.

Water-seal for Use when Boiling Liquids in a Reducing Atmosphere. H. Göckel. (*Zeits. angew. Chem.*, 1899, 620.)—This device is intended to be employed with flasks in which (for example) ferric iron solutions are being reduced by hydrogen. It preserves the liquid from contact with air; and, unlike the ordinary slit rubber valve, does not leave a high and dangerous vacuum within the vessel after its contents have been boiled and cooled. When inserted in the flask, the bulb is charged to just above the opening of the syphon tube with water or a solution of sodium bicarbonate; the main liquid can then be boiled briskly without driving the water out of the seal. When the flame is removed the bulb is filled up to the position shown in the diagram with a cold-saturated solution of sodium bicarbonate. As the pressure in the flask falls, liquid is sucked back through the syphon, which, on coming in contact with the acid, immediately liberates carbon dioxide, and soon restores and maintains the internal pressure equal with that of the atmosphere. In this manner, titration, etc., of the reduced solution can be safely postponed to a convenient opportunity.



F. H. L.

A Modification of the Sprengel Tube. A Minozzi. (*Gazzetta Chim. Ital.*, 1899, xxix., 406, 407.)—The tube shown in the accompanying figure was devised by the author as a convenient apparatus for determining the specific gravity of acids, while obviating all chance of losing any of the liquid by expansion on exposure to a higher temperature.



It holds about 50 c.c. from the mark on the capillary tube, *m*, to the extremity of the arm, *b*. In filling it the tubes *f* and *d* are fitted to *g* and *b* respectively, a mercury-pump being connected with *f*, while *d* is inserted through a cork pierced with two holes closing the bottle containing the acid.

The tube and the bottle of acid are both immersed in water at the required temperature, and when the liquid has been brought to the definite volume, the tube is closed by the stopper, *e*, and the cap, *c*. C. A. M.

MISCELLANEOUS.

WATER AND SEWAGE EXAMINATION RESULTS.

Report of the committee, consisting of Professor W. Ramsay (chairman), Dr. S. Rideal (secretary), Sir W. Crookes, Professor F. Clowes, Professor P. F. Frankland, and Professor R. Boyce, appointed to establish a Uniform System of recording the Results of the Chemical and Bacterial Examination of Water and Sewage.

The committee beg to report as follows: That it is desirable that results of analysis should be expressed in parts per 100,000, except in the case of dissolved gases, when these should be stated as cubic centimetres of gas at 0° C., and 760 millimetres in 1 litre of water. This method of recording results is in accordance with that suggested by the committee appointed in 1887 to confer with the committee of the American Association for the Advancement of Science, with a view to forming a uniform system of recording the results of water analysis.*

The committee suggest that in the case of all nitrogen compounds the results be expressed as parts of nitrogen over 100,000, including the ammonia expelled on boiling with alkaline permanganate, which should be termed albuminoid nitrogen. The nitrogen will therefore be returned as:

(1) Ammoniacal nitrogen from free and saline ammonia.

(2) Nitrous nitrogen from nitrites.

(3) Nitric nitrogen from nitrates.

(4) Organic nitrogen (either by Kjeldahl or by combustion, but the process used should be stated).

(5) Albuminoid nitrogen.

* British Association Report, 1889.

The total nitrogen of all kinds will be the sum of the first four determinations.

The committee are of opinion that the percentage of nitrogen oxidized—that is, the ratio of (2) and (3) to (1) and (4)—gives sometimes a useful measure of the stage of purification of a particular sample. The purification effected by a process will be measured by the amount of oxidized nitrogen as compared with the total amount of nitrogen existing in the crude sewage.

In raw sewage and in effluents containing suspended matter, it is also desirable to determine how much of the organic nitrogen is present in the suspended matter.

In sampling, the committee suggest that the bottles should be filled nearly completely with the liquid, only a small air-bubble being allowed to remain in the neck of the bottle. The time at which a sample is drawn, as well as the time at which its analysis is begun, should be noted. An effluent should be drawn to correspond as nearly as possible with the original sewage, and both it and the sewage should be taken in quantities proportional to the rate of flow when that varies (*e.g.*, in the emptying of a filter-bed).

In order to avoid the multiplication of analyses, the attendant at a sewage works (or any other person who draws the samples) might be provided with sets of twelve or twenty-four stoppered quarter-Winchester bottles, one of which should be filled every hour or every two hours, and on the label of each bottle the rate of flow at the time should be written. When the bottles reach the laboratory, quantities would be taken from each proportional to these rates of flow and mixed together, by which means a fair average sample for the twenty-four hours would be obtained.

The committee at present are unable to suggest a method of reporting bacterial results, including incubator tests, which is likely to be acceptable to all workers.

REVIEWS.

MILK, ITS NATURE AND COMPOSITION. By C. M. AIKMAN, M.A., D.Sc. London: A. and C. Black. Second edition, 1899, xxx., 180 pp.

The aim of this work is to give a short, popular statement of the more important facts of the chemistry and bacteriology of milk, and it is specially designed for students of agricultural science.

The work has been revised, but it is to be regretted that the revision has not been more thorough. On page 59 a correction has been made that the skin which forms on milk when heated consists chiefly of casein, and not, as is "erroneously supposed," of albumin; yet on page 171 the erroneous supposition is allowed to remain.

The manifest merits of the work—the able condensation into a small space of salient scientific facts, the lucidity and clearness of statement, and prominence of practical detail—are marred by faults and inaccuracies which a little more study and research and a little more care should have obviated. The most serious, from a practical point of view, is the statement that, working on a large scale, the separator yields about 93 per cent. of the fat in the cream; this means practically that separated

milk contains 0·25 per cent. of fat; if this quantity were found in a large dairy, it would mean either the dismissal of the man in charge as incompetent, or the replacement of the separator by one less faulty. Not more than 3 or 4 per cent. of the total fat should be found in the separated milk.

The analytical portion is very short, and working details are not given; the proportion of sulphuric acid used in the Leffmann-Beam method of fat estimation is given as double the proper amount.

Many of the faults are due to misuse of words; thus, "capronin" and "capronic acid" are used for "caproin" and "caproic acid," and "butin" (a word traceable to Heischmann) is given as a constituent of butter, but is found in no standard work on chemistry; "caseinogen" is used as synonymous with the "genuine" casein of Lehmann, and has not the significance assigned to it by Halliburton, who coined this name, and it is probably owing to the lax use of this word that the author appears to regard the product obtained by adding rennet to milk as identical, on page 29, with that produced by the action of acid, and as different on page 58.

We hardly expect in a work of this character to find the word "typhus" used to designate the disease known as "typhoid," or enteric fever. The late Ernest Hart certainly investigated no typhus epidemics, nor is it correct to speak of the Typhus bacillus. It is to be hoped that the word typhoid, to which the confusion is due, will soon cease to be an English word, and there will then be no chance of confusing enteric with typhus fever. H. D. R.

VICTOR VON RICHTER'S ORGANIC CHEMISTRY; OR, THE CHEMISTRY OF THE CARBON COMPOUNDS. Edited by Professor R. ANSCHÜTZ, University of Bonn. Authorized Translation by Professor EDGAR F. SMITH, University of Pennsylvania. Third American from the eighth German edition. Volume I.: Chemistry of the Aliphatic Series. Pp. 625. London: Kegan Paul, Trench, Trübner and Co., Ltd., 1899. Price 15s.

"Richter's Organic Chemistry" is certainly one of the standard works on the subject, and as such has had for many years a well-deserved and high reputation. It has passed through eight editions in German, and for English translations of the last three editions we are indebted to Professor E. F. Smith, of Pennsylvania. The latest edition is published in two volumes, edited by Professor R. Anschütz; the first of these deals with the aliphatic series, and the second (the English translation of which is not yet issued) with the aromatic series. From the first the work has been a comprehensive one, so much so, in fact, that the student acquainted with its contents might at least be said to have considerable acquaintance with the subject. It occupies an intermediate position between a text-book and a book of reference, yet the continual enlargement of successive editions must tend to bring it closer to works of the latter class. If, however, the lines on which it was originally drafted are to be preserved, the present enlargement of the text is fully justified by the important additions to our knowledge within recent years, and the advanced student will doubtless find it an invaluable *vade mecum*.

Apart, however, from the academical student, there are many professional chemists whose time does not allow them to follow closely all the latest researches

in specific branches of chemistry, but who nevertheless require to be posted up in the most recent facts and theories. To such, the present edition of "Richter's Organic Chemistry" will be of great service. It is quite unnecessary to plead the cause of organic chemistry, as it is self-obvious that something more than a mere superficial knowledge of that subject should be possessed by the modern analyst, who in his daily work is now called upon to separate and estimate various organic substances, and thanks to improved methods based on scientific principles, he is able to do this with an accuracy not inferior to that with which the constituents of a mineral may be estimated.

The subject-matter of the volume is well arranged, and the numerous paragraphs printed in smaller type will be found full of important supplementary information. Many references to original papers are given, and the editor seems to have exercised great skill in their selection. In the introductory portion, methods of elementary analysis and for the determination of molecular weights are described. The chemical constitution of carbon compounds is discussed, a particularly lucid exposition being given of the theory of molecular asymmetry, including the isomerism of optically active compounds and stereoisomerism, a branch of knowledge founded on the researches of Pasteur and developed by those of Van 't Hoff, Le Bel, von Baeyer, J. Wislicenius, and others. There are also remarks on the physical properties of carbon compounds and the action of heat, light and electricity on them. The general part calls for no additional remarks. The chemistry of the sugars, including the results of the remarkable researches of Emil Fischer, is dealt with in a plain and intelligible manner. Following a description of the simple sugars and their derivatives is a section headed "Carbohydrates," in which is to be noted the statement that these substances contain six, or a multiple of six, carbon atoms! But scant are the descriptions accorded to gums and other polysaccharides. The volume concludes with a few pages devoted to albuminous substances, etc. Under enzymes no mention is made of oxidizing enzymes, or of the enzyme of alcoholic fermentation, the latter phenomenon being attributed to an "organized ferment."

The whole work has doubtless profited by the fact that authorities in special fields of investigation have given the editor the benefit of their supervision of certain chapters. There appear to be no serious typographical errors, and considering the mass of detail brought together, this is certainly a circumstance which deserves commendation. Whilst, therefore, we are bound to admit that the translator has on the whole executed his task satisfactorily, there are certain paragraphs in which the diction is not the most elegant, and others where apparently too literal a rendering of the original text has been given. It is stated that "the presence of carbon in a substance is shown by its charring when ignited *away* from air." We read that "Michael offers the *thought*," etc. Carbocyclic compounds are referred to as "these ring-shaped bodies." What is doubtless in the original text expressed by the word "*Grenz-kohlenwasserstoffe*," is translated "limit hydrocarbons." There are also in one or two places cases of recapitulation, although as a rule this is avoided by cross-references. In conclusion, it may be worth mentioning that, although the volume bears the publishing date of the present year, the German edition appeared in 1898.

A. R. L.

THE ANALYST.

NOVEMBER, 1899.

PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS.

COFFEE EXTRACTS, THEIR COMPOSITION AND ANALYSIS.

By C. G. Moor and Martin Priest.

(*Read at the Meeting, June 7*, 1899.)

The various coffee extracts on the market show such remarkable variations in their composition that we thought it would be of interest to the members of this Society to record some figures recently obtained, and to invite discussion as to whether these articles ought to be dealt with under the Sale of Food and Drugs Act or not.

We give the figures obtained on 10 samples, 5 of which were sold as coffee extracts, 4 as coffee and chicory extracts, and 1 as coffee extract with sugar.

An exhaustive research on the composition of coffee extracts is to be found in the *Lancet* (special Analytical Sanitary Commission), July 7, 1894, in which figures are given on 14 samples of various kinds. In this paper it is stated that genuine coffee extracts contain about 0·5 per cent. of caffeine, and coffee and chicory extracts about 0·3 per cent.

Our figures are in fair agreement with these averages.

These very low caffeine figures are doubtless due to the unavoidable loss during manufacture, owing to the caffeine being precipitated as an insoluble tannate, which may sometimes be seen at the bottom of the bottle, and might (if a sample were divided into three parts) cause great variations in the composition of the three portions, unless great care is taken to thoroughly mix the contents. In some samples of coffee extract so much saccharine matter is added that the preparation is really a kind of treacle flavoured with coffee, and it is plain that Allen's comment (p. 553, Vol. III., part ii., "Commercial Organic Analysis") is correct, that "Coffee extracts are prepared with very limited success by subjecting roasted coffee to treatment with boiling water or steam, and adding the volatile products to the aqueous extract. The product is deficient in caffeine, and does not contain all the extractive matter of the coffee; nor when diluted with the appropriate amount of water is the colour the same as that of the freshly-prepared liquid."

The points which we suggest for discussion are these:

1. If sold as *coffee extract*, ought the article to contain a large amount of added saccharine matter unless its presence is declared?

2. As caffeine is probably the most valuable constituent of coffee, ought there not to be a minimum limit of caffeine for preparations of this class?

No.	Extract.	Ash.	N.	Caffeine.
1. Coffee extract	39·9	4·25	0·96	1·98
2. Coffee extract	27·9	0·95	0·15	0·47
3. Coffee extract and chicory ...	30·0	0·36	—	0·32
4. Coffee extract	34·8	1·28	0·23	0·54
5. Coffee extract	46·4	0·43	0·06	0·57
6. Coffee extract and chicory ...	37·6	0·36	—	0·02
7. Coffee extract	50·6	0·55	0·41	0·56
8. Coffee extract and chicory ...	48·6	1·87	0·37	0·26
9. Coffee extract and sugar ...	51·5	2·50	0·38	0·61
10. Coffee extract and chicory ...	48·5	1·14	0·30	0·28

Discussion.

The Chairman (Mr. Allen) said that the division of a sample of coffee extract into three parts would be rendered difficult by the fact that the caffeine and tannin separated out as a compound insoluble in cold water. Caffeine tannate, however, was easily soluble in hot water, as was well shown by an infusion of tea, which, on cooling down from a temperature at which it was perfectly limpid, became quite turbid, from the formation of a deposit of caffeine tannate. If, therefore, the task of division fell to the analyst, which in future it would not do, a fair sample could be obtained by simply warming up the extract before dividing it.

Mr. Hehner inquired whether the authors could explain the high percentage of caffeine in sample No. 1. It was about double that contained by coffee itself, and yet the total quantity of dry extract did not quite correspond with what would be expected if the sample were twice as concentrated as coffee.

Dr. Lewkowitsch said that caffeine was sometimes added in order to mask adulteration, as in the manufacture of artificial coffee-beans.

Mr. Cribb asked whether the authors had ascertained what the dry extract consisted of—whether, for instance, there was much caramel present.

Mr. Cassal said that to deal with coffee extract under the Sale of Food and Drugs Act as it at present stood would be a matter of very considerable difficulty. One of the first points would be to satisfy a bench as to the meaning of the term "extract." He would be glad to know in what way Mr. Moor would propose to arrive at a standard upon which a public analyst could work, and how he would propose to ascertain the degree of concentration of the extract, which he (Mr. Cassal) considered to be a necessary step before a certificate could be issued to the effect that a particular sample contained not less than so much treacle, or not more than so much coffee. Under existing conditions it would be very difficult to prove adulteration in the case of extracts. The points brought forward in the paper afforded additional evidence of the need of that Board of Reference, the establishment of which the Society had so strongly advocated.

Mr. Moor said that sample No. 1 had probably been enriched with caffeine, though this could not very well be ascertained. He had himself endeavoured to make coffee extract by boiling down *in vacuo*, but the caffeine separated out in such a way that it was impossible to prepare from the extract anything like a good imitation of coffee. The only one of these samples which at all resembled coffee was No. 1. He thought

(though from his own non-success in making an extract he could not be certain) that the caffeine present in this sample was in tolerably fair proportion to what would be obtained on boiling down an ordinary 10 per cent. infusion of coffee. This particular sample was not very highly coloured, but all the others no doubt contained a good deal of caramel. The excerpt quoted from Mr. Allen's book exactly represented the state of things at the present time. Coffee extract was a thing which ought not to be prepared at all. Its use was uneconomical, and in many cases it consisted of little else than treacle. If the division of samples was made by an inspector, it would be very unlikely that the proper proportion of caffeine would be contained in each third portion.

ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

FOODS AND DRUGS ANALYSIS.

The Question of the Retention of Phytosterin in the Animal System on Feeding with Cotton-seed Oil. C. Virchow. (*Zeit. für Untersuch. der Nahr. und Genussmittel*, 1899, ii., 559-575.)—The author was induced to undertake the experiments described in this paper by the interest which has been lately shown in the question of the adulteration of lard with cotton-seed oil. The experiments were made upon dogs and pigs. These were first reduced to a very lean condition by depriving them of food, after which they were given cotton-seed oil in conjunction with other food, and were finally slaughtered as soon as they had recovered their condition. The fat was then examined. The results entirely confirm the observations of A. Bömer on cholesterin and phytosterin, and show that phytosterin is not retained in the animal fat. H. H. B. S.

On the Digestibility of "Palmin" in the Human Alimentary Canal. H. Lührig. (*Zeit. für Untersuch. der Nahr. und Genussmittel*, 1899, ii., 622-632.)—"Palmin" is the trade name of a purified form of cocoanut-oil prepared by a secret process for use as an article of food. It is of a pure white colour, nearly free from water and acid, without granular structure, and of an agreeable nutty odour and taste. Cocoanut-oil being characterized by a relatively high saponification number, low iodine number, and high Reichert-Meissl number, it is easy to distinguish between a pure cocoanut-oil and a mixture of the butter with other fats. "Palmin" gave the following constants as the mean of several determinations:

	Melting Point.	Solidifying Point.	Iodine Number.	Saponification Number.	Reichert-Meissl Number.	Refraction at 40° C.
"Palmin"	25°	22·4	7·27	260·7	7·85	35·1
Insoluble Fatty Acids ...	25°-25·3°	22·8	6·90	271·1	—	18·1

A characteristic peculiarity of "palmin" is that it melts at 25° C., and then remains fluid below the melting-point until 19·8° C. is reached, when the thermometer rises rapidly, and remains between 22·3° and 22·4° C. until the fat has completely solidified again. The insoluble fatty acids of "palmin" also show the same peculiarity.

In two series of experiments, carried out upon the lines of the author's similar experiments with margarine and butter (*Zeit. für Untersuch. der Nahr. und Genussmittel*, ii., 484-506; ANALYST, 1899, xxiv., 237), it was found that from 95·5 to 97·3 of the fat was absorbed; hence the author concludes that the digestibility of "palmin" is not inferior to that of other foods. H. H. B. S.

Paraffin as an Adulterant of Oleomargarine. J. F. Geisler. (*Journ. Amer. Chem. Soc.*, vol. xxi. [7], pp. 605-608.)—The admixture of paraffin with oleomargarine appears to be on the increase of late, after a period of discontinuance subsequent to the revelations made in 1894, and the author has detected from 5 to 11·76 per cent. in samples recently examined. For its detection he recommends as a preliminary test a specific gravity determination; an abnormally low specific gravity of 0·9018 to 0·8907 at 100° F. (water at 100° F.), coupled with turbidity on saponification, are indications of the presence of paraffin. The solution will appear turbid if more than 2 to 3 per cent. of paraffin is present, and this can be collected for examination by careful alternate heating and cooling the solution. In one sample examined the fat—photomicrographs of which, showing irregular masses of paraffin, are given—did not melt clear below 106° F., congealing at 105° F., and yielding paraffin melting at 127°.

This adulteration is believed to be practised with the object of preventing the segregation of the solid and liquid fats in the mixture, and of imparting a texture resembling that of butter. C. S.

Studies on New Descriptions of Coffee. 1. Bourbon Coffee (Café Marron). T. F. Hanausek. (*Zeit. für Untersuch. der Nahr. und Genussmittel*, 1899, ii., 545-550.)—This coffee has already been noticed by H. Trillich, who published a description and analysis (*Zeit. öffentl. Chemie.*, 1898, 542), and by A. Froehner ("Die Gattung Coffea und ihre Arten," Dissertation Rostock. Leipzig, 1898). It is the product of a wild coffee-tree, probably *Coffea bourbonica*. The beans are egg-shaped, and thinner and more slender than true coffee-beans, as well as somewhat different in size. Pearl-beans are often found, and for these Trillich's description "tear-shaped" is peculiarly appropriate. The ordinary beans are from 10·5 to 14 mm. long, 6 to 7 mm. broad, and 2 to 4 mm. thick. The pearl-shaped beans are from 10 to 12 mm. long, 4·5 to 5 mm. broad, and 4 to 4·5 mm. thick. The roasted beans measure from 12 to 15·5 mm. in length, 7 to 8 mm. in breadth, and 3 to 5 mm. in thickness, thus showing considerable increase in size over that of the raw beans. The weight of the roasted beans is, however, 33·7 per cent. less than that of the raw beans, 100 weighing in the raw state 15·28 grammes, and roasted 10·22 grammes. By its size and shape alone, therefore, this coffee can be readily distinguished from Arabian coffee. Most of the beans remain enveloped in their

husks, which give to them a dull, greyish, dusty appearance. The husks can, however, be completely removed by steeping in water. Cut sections of the dry beans show that the husk is threefold, as the author has shown to be the case in Arabian coffee (*Zeit. für Nahrungsm.—Untersuch., Hygiene und Waarenkunde*, 1893, 85 and 105). Trillich's statement that caffeine is not a constituent of this coffee is confirmed by the author. The paper is illustrated by diagrams, showing the botanical structure of the beans. H. H. B. S.

Detection of Fluorine in Wine. G. Paris. (*Chem. Zeit.*, 1899, xxiii., 685.)—The ash from 50 c.c. of wine is mixed with a little precipitated silica and 0·5 or 1 c.c. of strong sulphuric acid in a small platinum crucible. The lid, bearing a drop of water suspended from its convex surface, and with its concavity filled with cold water (for condensing purposes), is then put in position, and the whole is heated for five minutes over a very small flame. After cooling, the drop of water is transferred to a microscope slide previously coated with Canada balsam, and 2 or 3 crystals of pure sodium chloride are added quickly. In twenty or thirty minutes the slide is examined with the microscope, using a power not exceeding 100 diameters. Under ordinary light, if fluorine is present, the hexagonal crystals of sodium hydrogen silico-fluoride will be recognised; while, under polarized light with crossed nicols, darkness is produced when the axes of the prisms lie parallel to the principal section of one of the nicols. The test is extremely sharp, and will detect an addition of 0·01 gramme of dry ammonium fluoride to 50 c.c. of wine. Employment of calcium chloride before incineration is not necessary. F. H. L.

The Composition of Edible Locusts. P. Rœser. (*Journ. Pharm. Chim.*, 1899, x., 203-206.)—In the north of Africa locusts are prepared for food by removing their head, legs, and wings, steeping them in 50 per cent. brine, and drying them in the sun.

Three specimens thus prepared have been examined by the author. They weighed from 0·55 gramme to 0·695 gramme, and had the following composition: Water, 9·1 to 10·4; organic matter, 79·05 to 81·00; mineral matter, 9·9 to 10·6; nitrogen, 7·0 to 8·4; fat, 12·2 to 16·6; chitin (insoluble in acids and alkalies), 5·4 to 7·9; chlorides as sodium chloride, 3·83 to 4·18; phosphoric acid, 1·059 to 1·087; and silica and sand, 2·10 to 2·40 per cent., together with calcium, potassium, iron, and sulphuric acid.

The fat, which was almost completely saponifiable, melted at 24·5° C.

C. A. M.

The Characteristics of Croton Oil. W. Dulière. (*Ann. de Pharm. de Louvain*, 1899, 229 and 278; through *Journ. Pharm. Chim.*, 1899, x., 305, 306.)—According to the author, oils prepared by extraction with petroleum spirit or by cold expression closely resemble the officinal oil, but oils obtained by hot expression, or by extracting the non-decorticated seeds with ether, differ from it in colour, acidity,

and degree of solubility in absolute alcohol, although their chief chemical constants are practically identical.

In the following table of these constants, the acetyl value was determined by Lewkowitsch's method, which the author has found to yield very concordant results (*Journ. Soc. Chem. Ind.*, 1897, 507):

Specific gravity at 15° C.	0·9437
,, ,, at 100° C.	0·8874
Solubility in alcohol (92 per cent.)	1 in 63
Critical temperature of dissolution (Crismer)	54·8° C.
Refraction (Zeiss degrees)	+77·5 at 27° C.
,, ,, (Amagat and Jean degrees)	+35 at 22° C.
Acid value (Bürstyn)...	21·8
Saponification value	215·6
Solidification point of fatty acids	16·4° to 16·7° C.
Reichert-Meissl value	12·1
Iodine value, after two hours	100·37 to 101·91
,, ,, twelve hours	103·63 to 104·39
,, of fatty acids (two hours)	111·23 to 111·76
Acetyl value	38·64

The presence of castor oil may be detected by converting the ricinoleic acid into sebacic acid, but the reaction, although sensitive, is not quantitative. The author states, however, that the difference between the acetyl values of croton and castor oils enables one to estimate very closely the amount of the latter.

In order to detect hydrocarbons, which it is stated are frequently introduced into croton oil, the sample should be distilled in a current of steam. Their amount may be calculated from the saponification value. C. A. M.

Detection of Synthetical [Toluene] Benzoic Acid in the Resin Sublimed Product. P. N. Raikow. (*Oesterr. Chem. Zeit.*, 1899, ii., 121.)—The recognised test for toluene benzoic acid in the material nominally prepared from gum benzoin depends on the assumption that the former is always contaminated with chlorine combined in some form with the aromatic nucleus; and the official method of carrying it out consists in igniting the acid with calcium carbonate. The author finds that his phloroglucinol-vanillin reaction (ANALYST, 1898, xxiii., 99) is a far more delicate means of detecting the presence of chlorine, and, as regards the truth of the dictum that chlorine indicates synthetical benzoic acid, he has instituted certain experiments. Benzoic acid sublimed from the best Siam benzoin or from inferior specimens of the gum is free from chlorine, therefore the occurrence of a red colour with phloroglucinol-vanillin proves the addition of acid from the toluene. On the other hand, whilst the majority of benzyl preparations made ultimately from toluene contain chlorine, *e.g.*, benzoic anhydride, benzylic alcohol, benzoyl acetate, benzonitrile, benzamide, benzaldehyde, mandelic acid, and methylic benzoate; others are free from chlorine, *e.g.*, benzylphenylhydrazine, the nitro- and amido- benzoic acids, benzylidenacetone, and ethylic benzoate; and it has not been possible to ascertain definitely whether the absence of halogen is due to the further processes involved in the manufacture of the latter bodies, or whether the original benzoic acid itself was free from

chlorine. Benzoic acid containing chlorine cannot be freed from this impurity by sublimation, or by crystallization from hot water or alcohol, consequently the failure of the above or the official test does not seem necessarily to prove that the synthetical acid is absent. F. H. L.

Hydrogen Peroxide as a Test for Salicylic Acid. W. E. Ridenour. (*Amer. Journ. Pharm.*, 1899, lxxi., 414-416.)—On adding to a solution of sodium salicylate a small amount of an ammoniacal solution of ammonium carbonate, followed by hydrogen peroxide solution, a colour reaction is obtained which varies from light pink or peach to dark garnet, according to the amount of salicylic acid present.

In the author's test experiments the strength of the sodium salicylate solution used was 10 per cent., and the hydrogen peroxide solution contained 2·30 volumes of available oxygen. The presence of the ammonium carbonate solution was found to be essential to the reaction, and 5 c.c. of the U.S.P. solution were used in each case. The weakest solution of sodium salicylate which gave the reaction contained 1 part of salicylic acid in 2,083 parts of water. When stronger solutions of hydrogen peroxide than that mentioned above were employed, the colour was produced at first, but rapidly disappeared.

The author adds a table showing the influence of other chemical substances on the reaction, the colours given by 0·5 gramme of the compound with 1 c.c. of the salicylate solution being summarized in the following list: Ammonium oxalate, none; ammonium chloride, cherry; sodium benzoate, cherry; sodium benzoate, cherry; sodium and potassium tartrate, dark cherry; sodium phosphate, cherry; hydrogen ammonium fluoride, none; sodium borate, amber; sodium thiosulphate, light lemon; ammonium sulphate, cherry; gallic acid, lemon; sodium acetate, cherry; sodium sulphite, none; potassium citrate, none; potassium chlorate, cherry; sodium hypophosphite, cherry; lactic acid (75 per cent.), cherry; tannin, yellow; potassium iodide, amethyst; potassium bromide, cherry; alcohol (15 c.c.), cherry; glycerin (15 c.c.), deep peach; and phenol, cherry. In most cases where no colour was obtained a reaction was given by using more of the sodium salicylate solution, sodium sulphite, however, being an exception.

With the exception of gallic and tannic acids, none of these substances gave any colour reaction by themselves with hydrogen peroxide and ammonium carbonate.

C. A. M.

TOXICOLOGICAL ANALYSIS.

The Detection and Estimation of Free Phosphorus in Oils and Fats. E. Louïse. (*Comptes Rend.*, 1899, cxxix., 394, 395.)—The methods used for the detection and estimation of phosphorus in toxicological work are not available when only traces of phosphorus are present in a large amount of oil. For it cannot be volatilized in a short time with vapour, whilst if the current be continued too long some of the phosphorus will be oxidized and left behind. Other methods based on the oxidation of the phosphorus are, in the author's opinion, also inapplicable under these circumstances.

This difficulty may be obviated by diluting the oil with acetone and precipitating the phosphorus by means of a concentrated solution of silver nitrate.

Five grammes of the oil are made up to 100 c.c. with acetone and the liquid divided into 10 portions. To the first of these is added 1 drop of a 10 per cent. solution of silver nitrate, the liquid filtered, and the filtrate again tested with silver nitrate. If a coloration be obtained, the second tube is tested with 2 drops of the silver nitrate solution in the same way, and so on until the point is reached at which the precipitation is complete. Say, for example, that 2 drops have been found insufficient and 3 too many. Two drops of the 10 per cent. solution are then added to a fresh portion and the intermediate point determined by means of a 1 per cent. solution of silver nitrate. In this way traces of phosphorus in oil can readily be determined in terms of standard silver nitrate. C. A. M.

ORGANIC ANALYSIS.

Influence of Temperature on the Specific Rotation of Sucrose, and Method of correcting Readings of Compensating Polarimeters. H. W. Wiley. (*Journ. Amer. Chem. Soc.*, vol. xxi. [7], pp. 568-596.)—From the results furnished by a number of carefully-performed experiments on the alterations in the specific rotation of sucrose under the influence of temperature, and on the various factors affecting the same, all of which are given in detail, the author concludes that the mean variation for each degree C. is 0·00994, the specific rotatory power at 17·5° C. being 66·547, whilst at 4° and 40° (the limits of temperature investigated) the readings were 66·657 and 66·299 respectively; *i.e.*, the variation is in inverse ratio to the temperature. It appears, however, that the variations are not uniform, the rate of decrease being greater (nearly double), between 15° and 17·5°; 20°-25° and 30°-35° C. than elsewhere in the series. C. S.

The Estimation of Mannose in the Presence of Other Sugars. E. Bourquelot and H. Herissey. (*Journ. Pharm. Chim.*, 1899, x., 206-209.)—The authors have devised a method for the estimation of mannose, founded on the insolubility of its hydrazone in cold water, which in its general principles is the same as that worked out by Lintner and Kröber for the estimation of dextrose, lævulose, and saccharose (ANALYST, xx., 167).

They find that the results are accurate if the temperature be kept sufficiently low, and the solution contain from 3 to 5 per cent. of mannose. With weaker solutions the weight of the osazone is too low by about 0·04 to 0·05 gramme for each 100 c.c. of solution. The method is stated to be specially applicable to the determination of mannose in the products of the hydrolysis of vegetable substances where the mannose is frequently accompanied by galactose, arabinose, and compounds allied to dextrins.

The following examples may be quoted to show the authors' method of working, and the results obtained in test experiments:

A solution of crystalline mannose (0·8 gramme) and galactose (1·20 grammes), in

33·2 c.c. of water, was mixed with a solution containing phenylhydrazine (2·4 c.c.) and glacial acetic acid (2·4 c.c.) in 12 c.c. of water. The mixture was left for eight hours at a temperature of 10° C., and the precipitated osazone washed successively, on a double filter with the aid of a pump, with 10 c.c. of ice-water, 10 c.c. of alcohol, and 10 c.c. of ether, dried at 100° C. and weighed. The amount of osazone found was 1·195 grammes, as compared with the theoretical quantity, 1·20 grammes.

In like manner a mixture of 0·8 grammes of mannose and 1·20 grammes of arabinose yielded 1·195 grammes of osazone; and a mixture of mannose (1 gramme) and crude maltose containing dextrin (1 gramme), yielded 1·44 gramme, as against the theoretical amount, 1·50 gramme. C. A. M.

The Determination of Albuminous Substances in Urine. G. Denigés. (*Journ. Pharm. Chim.*, 1899, x., 97-102.)—The method described here is an application of the author's method of estimating casein in milk (ANALYST, xxii., 11, and xxiii., 38). Although the weight of proteid precipitated is not proportional to the mercury combining with it, it is possible to make a correct estimation, when the amount of albumin in the urine required for the analysis (150 c.c.) does not exceed 15 to 16 centigrammes.

Twenty c.c. of the mercuric reagent are mixed with 2 c.c. of glacial acetic acid, and after the addition of 150 c.c. of the urine, which must not contain more than 1·10 gramme of albumin per litre, the liquid is made up to 200 c.c. and filtered. Of this filtrate 125 c.c. are added to 25 c.c. of an ammoniacal solution of potassium cyanide equivalent to $\frac{N}{20}$ silver nitrate, the liquid filtered after two or three minutes, and 120 c.c. of this second filtrate titrated with decinormal silver nitrate solution until a faint but permanent turbidity results. The number of tenths of a c.c. (n) required, less the constant 48, gives directly the number of decigrammes of albumin in a litre of the urine.

In the case of there being impurities in the reagent, the constant required may not be exactly 48. This should be determined by mixing 10 c.c. of the mercuric reagent with 20 c.c. of the cyanide solution and 100 c.c. of water, and adding decinormal silver nitrate until there is a permanent turbidity, the number of tenths of a c.c. used giving the necessary constant.

If the urine contain more than 1·10 gramme of albumin per litre, it must be so diluted that not more than that proportion is present.

The author's mercuric reagent is prepared by dissolving 13·55 grammes of mercuric chloride and 36 grammes of potassium iodide in 100 c.c. of cold water, and diluting the solution to a litre.

This method is not applicable when the urine contains less than 20 centigrammes per litre, and the author therefore recommends the following process in such cases: An albuminous urine, the albumin in which has been previously determined gravimetrically, is distributed throughout a series of five tubes in such a manner that they contain albumin in the proportion of 1, 2, 4, 8 and 12 centigrammes per litre respectively. The liquid in each is diluted to 10 c.c., and 10 c.c. of the urine under examination is placed in a sixth tube. After the introduction of 2 c.c. of a 5 per cent.

solution of sodium metaphosphate, and 4 drops of sulphuric acid into each, the tubes are placed for five minutes in boiling water, and the opalescence in the sixth tube compared with that in the standard tubes.

This method gives good results with urine containing not more than 12 centigrammes of albumin per litre, but above that quantity the precipitate formed in the tubes on warming is of a flocculent character. Therefore, in the examination of urines containing between 12 and 20 centigrammes per litre there must be a suitable dilution.

When globulins are present in the urine, the total albuminous substances are estimated as described above, before and after saturation with magnesium sulphate, which removes the globulins. Two determinations are also necessary in the presence of alkaloids or peptones, viz., before and after the removal of coagulable albuminous substances by heat. C. A. M.

A Reaction for Benzidine and Tolidine. J. Wolff. (*Ann. de Chim. Anal.*, 1899, iv., 263-264.)—A small portion of the substance under examination is dissolved in 1 c.c. of cold glacial acetic acid, the test-tube filled to about three-quarters with water, and a few drops of water, with lead dioxide in suspension, introduced. A bright blue colour is immediately produced, which lasts for some time, but disappears on heating.

If to the solution of either of these bases in dilute acetic acid a few drops of bromine be added, there is a blue coloration, and on continuing the addition of bromine a blue precipitate is formed. The colouring matter thus obtained is stable at the ordinary temperature; it is insoluble in water and in alcohol. The colour disappears on adding ammonia, but reappears on again acidifying with acetic acid. In the presence of dilute mineral acids the oxidation of benzidine or tolidine does not give any blue coloration. The colour is obtained, though with less intensity, in the presence of dilute solutions of other organic acids such as citric, tartaric, and oxalic acids. C. A. M.

Determination by Combustion of Carbon and Hydrogen in Organic Substances containing Nitrogen. O. F. Tower. (*Journ. Amer. Chem. Soc.*, vol. xxi. [7], pp. 596-605.)—From the results obtained in the investigations described, it appears that, in the case of amido-compounds, there is no need to use a copper spiral in the combustion-tube, since no oxides of nitrogen absorbed by concentrated sulphuric acid or by soda-lime are formed, whilst of the nitro-substances (nitraniline, dinitrobenzene, and picric acid) examined, only the last-named gave off oxides—probably for the most part nitrogen peroxide—absorbed by the reagents. The formation of nitric oxide is not impossible, but as it is not absorbed unless further oxidized, it does not affect the accuracy of the determination of carbon and hydrogen. C. S.

Notes on Acetone Oil. **R. Duchemin.** (*Bull. Soc. Chim.*, 1899, xxi., 798-800.)—The author supplements the work of A. and P. Buisine (ANALYST, xxiv., 214), which dealt principally with suint oils, by analyses and remarks on the composition of pyrolignite oils. These are much less regular as regards the percentage of the various constituents. Thus, two samples contained about 23 per cent. of substances distilling below 90° C., and from 60 to 66 per cent. distilling between 90° and 110° C.; while two other samples of different origin contained only 30 to 35 per cent. of the former, and 27 to 35 per cent. of the latter constituents.

The following analyses are given to show the composition of three samples of acetone oil manufactured from gray acetate of lime, which the author found to contain only traces of propionates—a fact which accounts for the almost complete absence of methyl isopropyl-ketone and methyl propyl-ketone in the distillation products. The third sample was washed several times, as is customary in most French factories:

I. Distilling between ° C.	Per Cent.		II. Distilling between ° C.	Per Cent.		III. Distilling between ° C.	Per Cent.	
63– 70	20		18			70– 80	5	70° to 90°, 18 per cent.
70– 75	19	70° to 90° 51 per cent.	17	70° to 90°, 41 per cent.		80– 85	4	
75– 80	15		10			85– 90	9	
80– 85	12		10			90–110	2	
85– 90	5		4			Above 110	80	
90–110	2		2					
Above 110	27		39				100	
	100		100			Insoluble in water ...	70	
Insoluble in water	42					Insoluble in bisulphite ...	76	
Insoluble in bisulphite solution	35		48 33					

The variations in composition of commercial pyrolignite acetones are therefore to be attributed partly to difference in the composition of the calcium acetate and partly to the amount of washing which the acetone has received. According to the author, these oils can only contain traces of methyl propyl-ketone, but may contain up to 50 per cent. of their weight of products distilling below 90° C., and in particular of methyl ethyl-ketone. C. A. M.

On Hazel-nut Oil. **J. Hanus.** (*Zeit. für Untersuch. der Nahr. und Genussmittel*, 1899, ii., 617-622.)—This oil is contained in the hazel-nut (*Coryllus avellana L.*) to the extent of from 50 to 60 per cent. In the following table the constants found by the author are given side by side with those already published by other chemists, for comparison:

	Hanus.	Schädler.	Filsinger.	De Negri and Fabris.	Soltsien.	Schöttler.
I. Natural oil:						
Specific gravity at 15° C. ...	0·9169	0·9243	0·9146	0·917	0·9164	0·916
Saponification number ...	193·7	—	197·1	192·8	191·4	187·0
Iodine number	90·20	—	—	86·3-86·9	83·2	87·0
Hehner number	95·60	—	—	—	—	95·50
Reichert-Meissl number ...	0·99	—	—	—	—	0·99
Acetyl number	3·2	—	—	—	—	—
Glycerin per cent. ...	10·41	—	—	—	—	—
II. Insoluble fatty acids:						
Saponification number ...	200·6	--	—	—	—	—
Iodine number	90·60	—	—	—	—	—
Mean molecular weight ...	279·0	—	—	—	—	—
III. Unsaturated fatty acids:						
Saponification number ...	198·50	—	—	—	—	—
Iodine number	91·30	—	—	—	—	—
Mean molecular weight ...	282·0	—	—	—	—	—
Maumené number	36·2	—	—	—	—	—

The results of the analysis of the oil are as follows: Oleic acid 85 per cent., palmitic and stearic acids 10 per cent., glycerin (calculated from the saponification number) 10·41 per cent., and phytosterin 0·50 per cent. H. H. B. S.

The Examination of Wood-Tar Creosote. L. F. Kebler. (*Amer. Journ. Pharm.*, 1899, lxxi., 409-413.)—The composition of commercial creosote varies with the kind of wood employed for the distillation, and the methods employed for removing the creosote from the distillate. It consists of a mixture of phenoloid compounds, chiefly of several homologous series, and any of the following compounds may be met with: Phenol (boiling-point, 182° C.); paracresol (203° C.); xylol (220° C.); guaiacol (200° C.); cresol (219° C.); homocresol, or dimethyl-guaiacol (230° C.); and cœrulignol, or propyl-guaiacol (241° C.).

Wood-tar creosote was formerly regarded as consisting in the main of guaiacol and cresol, but at the present time the author states that it is difficult to purchase creosote containing more than 20 per cent. of guaiacol; and he regards the high percentage found by some chemists as being probably due to faulty methods of analysis.

The differentiation of different kinds of creosote is uncertain. Hirschsohn (*Pharm. Zeit. Russ.*, 1898, xxxv., 801) has described tests to distinguish beech-wood tar from the tars of birch, fir, and juniper; but, in the author's experience, the tests which will identify the unmixed products are uncertain in the case of mixtures.

Oak-wood creosote is much more caustic than beech-wood creosote, owing to the fact that the former contains a larger amount of monophenols and correspondingly less guaiacol than the latter. The proportion of cresol and its homologues is the same in both.

Pine-wood creosote (*Comptes Rend.*, cxix., 1276) distilling between 200° and 220° C., has been found to contain 40 per cent. of monophenols, 20·3 per cent. of guaiacol, and 37·5 per cent. of cresol and its homologues.

As regards the specific gravity of creosote, the United States Pharmacopœia fixes the lowest limit at 1·070, at 15° C.; while the British Pharmacopœia requires it to be not lower than 1·079, at 15° C. According to the author, the former limit can be readily met by a creosote which does not contain any guaiacol, and in his opinion the American standard should be raised.

The following analyses of six samples from different American makers are given to show that it is possible to judge as to the quality of a creosote by an analytical examination:

	Specific Gravity.	Boiling Point, ° C.	Per Cent. of Substance distilling between the following Temperatures. ° C. corrected.					
			1–200°.	200°–205°.	205°–210°.	210°–215°.	215°–220°.	220°–235°.
1	1·0748	195–224	5	34	26	23	6	3
2	1·0748	195–222	20	20	30	19	7	1
3	1·0650	210–238	0	0	0	30	25	40
4	1·0642	208–238	0	0	2	37	21	36
5	1·049	188–220	18	12	12	39	14	—
6	1·069	200–225	32	18	10	24	9	4

	Colour.	Reaction.	No. of c.c. of 7·5 per cent. NaOH required to dissolve 2 c.c. of Creosote.	Glycerin Water Test.	Twenty c.c. of Alcoholic KOH mixed with 1 c.c. of Creosote.	Guaiacol, Per Cent.	Potassium Guaiacol and Cresol, Per Cent.
1	Nearly colourless	Faintly acid	9	Normal	Crystals in 15 minutes; solid in 40 minutes	None	60
2	Nearly colourless	,,	8	,,	No crystals in 5 hours; solid in 18 hours	None	48
3	Amber	,,	9	Emulsion	Crystals in 4 minutes; solid in 15 minutes	8	60
4	Straw colour	Neutral	7	,,	Solid almost immediately	16	106
5	Nearly colourless	,,	8	,,	Solid on cooling	—	—
6	Nearly colourless	Faintly acid	8	Normal	Solid on cooling	—	—

The author points out that none of the samples complied with the requirements of the United States Pharmacopœia.

For the estimation of the guaiacol, the following process was used: 5 c.c. of the creosote are mixed with 50 c.c. of a 20 per cent. alcoholic solution of potassium hydroxide, and the resulting crystalline compounds of guaiacol and cresol, which deposit in from ten to thirty minutes, are pressed between filter-paper until dry. The mass is then mixed with 5 c.c. of 10 per cent. sulphuric acid, and the mixture heated for a moment until the guaiacol and cresol rise to the surface. Water is then added until the oily layer sinks to the bottom, the aqueous layer decanted off, and 4 c.c. of concentrated ammonium hydroxide added. The guaiacol immediately forms a hard crystalline compound, and after some time a semi-crystalline mass results from the combination of the cresol. On treating the mixed compounds with benzene (benzin), only the ammonium compound of guaiacol remains undissolved, and this can be

separated by decantation or filtration and washed. Finally, the residue is rendered acid with 10 per cent. sulphuric acid, and the guaiacol extracted with benzene, evaporated in a weighed basin, and weighed.

In order to differentiate creosote from coal-tar phenols, one volume of the sample is shaken with a mixture of glycerin and water (3 : 1), and left to separate. The diminution in the volume of creosote indicates approximately the amount of soluble impurities.

The barium hydroxide test for cœrulignol and other high-boiling constituents gave a negative result with each of the six samples.

In the author's opinion, the pharmacopœial requirements of creosote should be based on the following points: Physical appearance, reaction, solubility, specific gravity (not lower than 1·080, at 15° C.), boiling-point (200° to 220° C.), reaction with a 20 per cent. solution of potassium hydroxide in absolute alcohol, and a test for neutral oils. C. A. M.

The Characteristics of the Oil and Terpenes of Aralia Nudicaulis. W. C. Alpers. (*Amer. Journ. Pharm.*, 1899, lxxi., 370-378.)—The rhizomes used by the author contained from 40 to 60 per cent. of water, and after being dried at 100° C., yielded on the average 5·53 per cent. of ash, of which nearly a quarter consisted of chlorides and sulphates of sodium and potassium.

The volatile oil which was obtained from 50 kilos of the fresh roots by distillation with steam, and extraction of the essential oil from the distillate with chloroform, amounted to from 0·04 to 0·12 per cent.

It boiled at 260° to 270° C. under ordinary pressure, whilst the larger proportion of it distilled at 185° to 195° C. under a pressure of 88 mm.

It was found to consist principally of a sesquiterpene, $C_{15}H_{24}$, and an alcohol, $C_{15}H_{25}\cdot OH$(?), with a small quantity of a clear blue oil distilling at about 300° C., probably azulene $C_{16}H_{26}O$ (B.P. 302° C.).

The sesquiterpene had a specific gravity of 0·9086 at 20° C., boiled at 270° C., and had a specific rotation of $[\alpha]_D = -7$ to $-8°$. It formed oily addition-compounds with hydrochloric acid and bromine, and derivatives with nitrous acid.

When dissolved in three parts of glacial acetic acid, and mixed with an equal amount of glacial acetic acid previously saturated with dry hydrochloric acid, it gave a rose colour, which gradually became purple, and subsequently, in about ten minutes, sky-blue. The latter colour was permanent, and the liquid on distillation *in vacuo* yielded a blue liquid at about 140° C.

Other colour-reactions were obtained by dissolving it in chloroform and adding sulphuric acid, which produced a purple-red coloration, and by adding sulphuric acid to its solution in acetic acid which gave a light wine-red colour.

As these reactions and properties distinguish this sesquiterpene from isomeric compounds previously described, the author proposes for it the name *araliene*.

C. A. M.

The Composition of Colophony. R. Henriques. (*Chem. Rev. Fett. u. Harz. Ind.*, 1899, vi., 106-111.)—The work described in this paper was undertaken with the

object of determining whether the ester value of colophony was due to the presence of esters, and if not, what class of compounds were present.

Dark samples of colophony were used in the experiments, since these have a greater ester value than lighter specimens. Cold saponification was employed, as it was found that with hot saponification there was evidently a more deep-seated change.

The saponification value was determined by treating about 2 grammes of the substance with semi-normal alcoholic alkali, shaking for ten minutes until clear, and leaving the solution for about sixteen hours before titrating. Concordant results were obtained in this way, but the author considers that the results (about 3 units higher) obtained by leaving the liquid for forty hours, or by using normal alkali and leaving for 16 hours, may possibly be nearer the truth.

Dieterich concluded that there were no esters in colophony, since he was unable to isolate alcoholic bodies from the products of saponification, but the contrary is asserted by R. Schick (*Zeits. angew. Chem.*, 1899, 28) and others.

The author reasons that, if the ester value be due to the decomposition of esters, a new quantity of acids must result on saponification, so that if after complete saponification the resin acids be liberated, washed, and their acid value determined and calculated on the original amount of substance taken, this second acid value will be greater than the first, provided that there has been a decomposition of esters. Otherwise the two values will be identical or nearly so. In practical experiments they were found to agree within about a unit, and hence the author concluded that there could have been no decomposition of esters.

As considerably higher saponification values were obtained by using normal alkali and leaving the solution for forty hours, it was considered possible that the alkali acted on the acids, producing soluble acids of lower molecular weight. But since in one specimen the acid value was raised by saponification, but not raised in the case of another, the author regarded the question as open.

As it was possible that the constant ester value of colophony might be due to the presence of lactones, attempts were made to isolate such bodies, but they were not found at all, or at most in small amount (1 to 1·5 per cent.), the main constituents being resin acids and unsaponifiable matter (8 to 12 per cent.).

Finally, the conclusion was arrived at that part of the resin acids themselves were probably of a lactonic character, containing both carboxyl groups and anhydride groups, and forming, in addition to the alkali salts obtained by direct titration, higher basic salts on exposure to the action of alkali in excess.

In the author's opinion there is nothing to justify the view of Dieterich that the slow saponification of colophony is due to the presence of acids so weak that they can only combine with alkali gradually and after a long time. As opposed to this view, he points out that the whole of the colophony acids are soluble in a cold aqueous solution of sodium carbonate, and that if colophony be treated with a moderately concentrated cold solution of sodium hydroxide, ether extracts little but unsaponifiable matter from the liquid.

As there is a relationship between oxy-acids and lactone acids, and also a certain degree of proportion between the amount of the ester value and the insolubility of the

acids in petroleum spirit, the author has applied Fahrion's method of estimating oxy-acids to the separation of the lactone acids (ANALYST, xxiii., 275).

Fifty grammes of colophony (saponification value, 189·8; acid value, 158·7) were left overnight in contact with four or five times the volume of petroleum spirit. The supernatant liquid was decanted, and the residue boiled with four successive portions of petroleum spirit, dissolved in a hot solution of sodium hydroxide, cooled and filtered. The filtrate was acidified, and the resin acids filtered off, washed and dried. They were then pulverized, again boiled with petroleum spirit, dissolved in ether, the solution filtered, the solvent evaporated, and the acids dried at 100° C. In two determinations with the same sample of colophony they amounted to 36 and 37·2 per cent. respectively.

The resin acids soluble in petroleum spirit were of a light yellow colour, resembling that of the finer kinds of colophony. Their acid value was 164·4, and their ester value 4·0 to 4·8. By further treatment with petroleum spirit, this ester value was reduced still more.

The insoluble acids were much darker, and on treatment with alkali yielded an intense reddish-brown solution. Their saponification value was about 196, their acid value 151·3 to 151·7, and their second acid value 144·8 to 147·6. Under the influence of hot saponification these values were greatly increased and not concordant, saponification values of 274 and 324, and a second acid value of 317·8, being obtained.

A further proof of the greater decomposition effected by hot saponification was shown by the fact that the recovered acids were no longer completely insoluble in petroleum spirit, and, unlike those from the cold saponification, gave an intense yellow colour on treatment with that solvent.

As additional evidence that the constant ester value of the acids insoluble in petroleum spirit was due to the hydrolysis of the lactone groups, these acids were converted into calcium salts before and after saponification, and the amount of calcium in each determined. This was found to be 7·11 per cent. before and 9·30 per cent. after saponification.

The author has not yet determined the exact point at which this decomposition of the lactone groups is complete.

The well-known Storch's reaction with acetic anhydride and sulphuric acid was given by the insoluble acids as readily as by the soluble acids.

It was found that the light yellow varieties of colophony also contain traces of the lactone acids insoluble in petroleum spirit. In one experiment the author isolated 0·4 per cent. with the same characteristics as that obtained from the darker specimens.

From these experiments the following conclusions were finally arrived at:

1. Colophony does not contain esters.

2. Acid anhydrides are not present in any quantity, but in the main unsaponifiable matter and free resin acids.

3. These acids can be separated by means of petroleum spirit into normal soluble acids and insoluble acids having the character of lactone acids.

4. The soluble acids have only an acid value, whilst the insoluble acids have a high constant ester value in addition to their acid value. C. A. M.

Analysis of Indiarubber Articles. R. Henriques. (*Zeits. angew. Chem.*, 1899, 802.)—The author's processes, which were originally described in the *Chem. Zeit.*, 1892, xvi., 1595; 1893, xvii., 707; 1894, xviii., 411 and 905 (ANALYST, xviii. 13, 227; xix. 111; xxii. 134), have stood the test of time; but, having been altered in certain particulars, are worth recording anew.

(1) *Estimation of total Sulphur and Metals.*—A 6 cm. hemispherical porcelain basin holding 30 c.c. is charged with 10 c.c. of pure strong nitric acid, covered with a watch-glass, and placed on a warm water-bath. 1 gramme of the sample (rasped or cut up) is weighed off, and a fragment dropped into the acid; as soon as red vapours appear, the rest is cautiously added at such speed that the reaction is continuous without becoming stormy—it can be controlled by removing the basin from the water-bath, or by heating it more strongly. When everything is dissolved, the cover-glass is wiped with a few pieces of paper which are dropped into the liquid, the whole is evaporated to a syrup, 20 c.c. more nitric acid are introduced, and the evaporation is repeated till most of the free acid is driven off. The mass is then stirred up with a mixture of 5 parts of sodium carbonate and 3 parts of potassium nitrate, covered with a layer of the same (using about 5 grammes altogether), warming, if necessary, on the water-bath till all carbon dioxide is evolved. The basin is then covered with a similar vessel, concave side down, and heated with great care and very slowly over a minute flame, which is gradually urged till the mass becomes black. The temperature is next raised until the salts fuse, the upper basin being removed towards the end of the operation. If successful, the cover only contains brown products of distillation which are free from sulphur; but if spitting has occurred, some particles may adhere to the upper basin, and must be separately fused with the saline mixture. Finally, the melt is extracted with boiling water and filtered; the solution is acidified and silica and sulphuric acid determined; the insoluble matter is taken up with warm hydrochloric acid to separate silica from the metals, which are then severally estimated as usual. In the case of rubber goods containing cinnabar a separate portion is taken, which, after decomposition with nitric acid, must be treated with water, and in the filtrate the heavy metals must be thrown down with sulphuretted hydrogen.

(2) *Estimation of Rubber Substitutes in Vulcanised Products.*—This process, though tedious, has not been found capable of simplification; it remains as outlined in the *Chem. Zeit.*, 1892, 1625, and 1894, 411 (ANALYST, as cited above). A small improvement consists in removing the partially-dried caoutchouc mass from the filter, drying and weighing it separately, instead of employing a tared paper and weighing rubber and paper together.

(3) *Estimation of Unsaponifiable Oils in Vulcanised Products.*—Mineral oils, rosin oil, and paraffin are to be found in many varieties of rubber goods; especially such as have been worked up a second time. After the mass has been treated with alkali, dried and weighed, the product is rubbed out in a basin and extracted with ether as long as the solvent becomes coloured; the solution is simply poured off, the residue is dried again and weighed, the loss being unsaponifiable oils. Ash and combined sulphur are determined on the product freed from oil.

(4) *Estimation of Rubber Substitutes in Non-vulcanised Products.*—Although suited for the examination of non-vulcanised "patent" rubber plates, the regular process is not available for the treatment of sheets composed of soft rubber and substitutes; the difficulty, however, is only due to physical causes, and the following modification avoids it: 5 grammes of the sample are boiled under an inverted condenser with 25 c.c. of benzene for an hour, and allowed to rest over-night. 25 c.c. of normal alcoholic alkali are added, and the boiling is repeated for four hours. The solvents are removed on the water-bath, and the residue is rinsed into a large basin with hot water. It is extracted with boiling water several times, the alkaline liquor being simply poured away; the rubber that remains is kneaded and boiled till no longer alkaline, and it is finally dried to constant weight at 100° C.

(5) *Estimation of Unsaponifiable Oils in Non-vulcanised Products.*—The ether process described in (3) obviously cannot be employed in the present instance; hot acetone is used similarly. But as the residual rubber is not easily brought into a condition fit for weighing, the extracts are run into a flask and the acetone distilled off; the oils are taken up in ether, filtered into a tared beaker, and themselves dried and weighed.

(6) *Estimation of Carbon Dioxide.*—Chalk, magnesium carbonate, white-lead, and zinc carbonate are common loading materials; but as the respective oxides are sometimes employed in their stead, it is often necessary to determine the carbon dioxide. Vulcanised goods are finely rasped and treated in Geissler's apparatus, charging the reaction-flask with dilute copper sulphate solution in place of water, as sulphides are frequently present. From non-vulcanised goods, the rubber must be partly or wholly removed by boiling with nitrobenzene, and the residue powdered and examined as before.

Henriques remarks that he is unable to obtain uniform satisfactory results with Weber's solvent for indiarubber (nitrobenzene). Non-vulcanised materials, and such as are only lightly vulcanised and loaded, dissolve readily; but neither hard rubber goods, nor soft and heavily-loaded articles, can be completely separated therewith; the liquids yielded by the latter class of wares, moreover, are exceedingly difficult to filter. F. H. L.

INORGANIC ANALYSIS.

Modification of Pierce's Method for the Determination of Arsenic in Ores. J. F. Bennett, junr. (*Journ. Amer. Chem. Soc.*, vol. xxi. [5], pp. 431-435.)—The original method being rendered inaccurate by the solubility of silver arsenate in an excess of acid or alkali and in ammonium nitrate, by the extreme fineness of the precipitate and by the inadequacy of litmus as an indicator, the author proposes the following modification:

One-half gramme of the finely-powdered substance is fused with 3 to 5 grammes of sodium carbonate and potassium nitrate in equal parts, about one-third being used as a cover. On cooling, the mass is extracted with boiling water and filtered. The filtrate, which contains the arsenic as alkali arsenate, is strongly acidified with acetic

acid, then boiled to expel carbon dioxide, and, after cooling, treated with a few drops of alcoholic phenolphthalein and sufficient sodium hydroxide to give an alkaline reaction, the purple-red coloration produced by an excess of alkali being discharged by acetic acid. A slight excess of neutral silver nitrate is then well stirred in, and the whole left to settle, away from direct sunlight; the supernatant liquid is poured off through a filter, and the precipitate washed by decantation with cold water, thrown on the filter and washed thoroughly. The funnel being then filled with water and 20 c.c. of strong nitric acid, this liquid is run through the filter into the original beaker, the residue washed thoroughly with cold water, and the filtrate made up to about 100 c.c. and titrated with standard potassium thiocyanate (Volhard); or the silver is determined by scorifying the filter and precipitate, and cupelling, the first method being adopted when chlorides are present. C. S.

Valuation of Gold Samples. W. Witter. (*Chem. Zeit.*, 1899, xxiii., 522.)—A short time ago Bock stated that the ordinary process of gold cupellation was no longer delicate enough for modern requirements, and he propounded a new method of investigation based partly on electrolysis (*Chem. Zeit.*, 1897, xxi., 973; 1898, xxii., 358). The present article describes experiments carried out at the request of the Hamburg Mint to ascertain how great is the difference between the results of wet analysis and dry assay, whether assay is accurate enough to govern large financial transactions, whether Bock's process is better than cupellation, and whether it is always applicable. Starting with four carefully-analysed samples of gold—an unmelted electrolytic product, commercial electrolytic fine gold, washed gold as sent from Hong-Kong, and gold coinage (900°)—using always the same quality acids, lead, and cupels, and the London Mint system of treating the rolls (Rose, "Metallurgy of Gold," 1896, 445), the author found, as first enunciated by Rose, that the temperature of the muffle is a most important, though often overlooked, factor in obtaining correct figures by cupellation. Employing Heraeus's platinum-rhodium element with a Keiser and Schmidt galvanometer, he deduced the following as the proper working temperatures: Chemically pure gold, 960° C.; less fine gold, 950° to 960° C.; gold containing a very little platinum, 1000° to 1010° C.; 973° malleable gold containing silver and copper, 960° C.; coinage metal (900 parts Au, 100 parts Cu), not exceeding 920° or 930° C.; 720° gold with silver and copper, below 900° C. The detailed figures show also that when an approximately pure gold, such as an electrolytic product, comes to be assayed, the result of the test cannot be checked on absolutely pure metal; because (presumably) of the platinum in the former, which demands a higher temperature. Therefore the temperature of cupellation must be determined; and when a good pyrometer is not at hand, a series of alloys with melting-points rising by 20° or 25° between 850° and 1050° C., should be provided as guides. Analyses of the rolls obtained from the four varieties of metal mentioned above proved them to contain from 99·8576 to 99·8786 per cent. of gold, while the cupels retained 0·0370 to 0·0891 per cent.; the difference between the weights of the rolls and the true gold in them ranged between 0·1226 and 0·1424 per cent.; therefore the proportion of gold volatilised was between 0·0335 and

0·0890 per cent. These results fairly bear out Napier's statement that, according to the composition of the material, the difference between the amount of gold in the sample and in the roll (as determined by analysis) varies between 0·12 and 0·14 per cent. It is thus necessary to watch that the weight of foreign metals in the rolls shall equal the weight of gold volatilised; but that this is practically possible the author's experiments demonstrate. In short, if the temperature be kept at the proper point, the yield of the cupellation process will not differ from the theoretical by more than 0·2 part per 1000.

Now, especially with high-grade samples, a wet analysis cannot safely be conducted by determining directly the amount of gold; for, as Krüss's researches have shown (Liebig's *Annalen*, 1887, ccxxxviii., 67), precipitation of pure gold chloride may lead to errors ranging between −0·4 or −1·4 part to +0·3 part per 1000. Thus, the foreign metals must be estimated, a process which requires at least four weeks, and quantities of 50 or 100 grammes for its successful operation. Very low quality gold cannot be assayed, and analysis becomes necessary; but such specimens are rarely met with at the present time. Gold is usually sold in parcels of 5 to 25 kilos., each of which is tested; therefore the sum in dispute, assuming the two tests to differ by the maximum amount (0·4 part per 1000), is comparatively small; and owing to the greater rapidity of assay, the interest on this sum is less than when a wet analysis is insisted on. On the whole, if properly conducted, cupellation yields quite satisfactory results, and an analysis is seldom of practical value.

In Bock's process the sample of gold is melted with silver in a graphite crucible, the product rolled out, melted again, once more rolled, and then treated as usual with nitric acid of the specific gravities 1·2 and 1·3. The rolls are next electrolysed for ten minutes, washed, etc., and ignited. Bock omitted to specify the exact strength of the current; Witter finds as the maximum to avoid danger of disintegration, 1 ampère (ND_{100}) calculated on the cathode, using 1 part of 1·2 nitric acid and 5 parts of water as the electrolyte. It seems better, however, to allow the current to pass for twenty or thirty minutes at a density of less than 1 ampère (0·05 to 1·0) in order that all silver, and all other metals that are removable, shall be dissolved. Nevertheless, the whole of the silver and other metals cannot always be removed, and it is sometimes necessary to repeat the process; on the contrary, losses may occur in the crucible and by disintegration during boiling or during electrolysis. As the result of a large number of trials, Bock's method appears to give average errors of +0 to +0·2 part per 1000. Bock's process has the advantage of not needing so much previous experience to bring it to a successful issue, and of leaving cupellation available as a check; but it is not suited to all varieties of impure gold. Primarily intended for coinage metal, it can also be employed on any silver and copper alloys that are malleable; brittle alloys, by simple fusion with silver, still remain brittle, and so they cannot be beaten or rolled out ready for the electrolytic treatment. In laboratories where frequent analyses of a uniform malleable quality of gold have to be made, Bock's process is to be recommended; but in places where the samples vary widely, a preliminary dry assay is indispensable, and it depends entirely upon circumstances whether the electrolytic method is applicable to control the results. F. H. L.

Determination of Gold, and its Separation from Platinum and Iridium. L. Vanino and L. Seeman. (*Ber.*, 1899, xxxii., 1968.)—When a solution of gold chloride is mixed with hydrogen peroxide and sodium (or potassium) hydroxide, the metal is completely thrown down in the cold within the space of a few minutes; whereas precipitation with ferrous sulphate or oxalic acid requires several hours. So delicate is the reaction that it will detect 0·03 milligramme of gold in 10 c.c. of a liquid, the other tests beginning to fail between 0·09 and 0·06 milligramme per 10 c.c. A test with ferric chloride, hydrochloric acid, and a fragment of zinc, stated by its author to be capable of detecting 1 part of gold per 1,000,000 (*Pharm. C. H.*, xxvii., 321), is equally sensitive with the present one, giving a reddish coloration in fifteen minutes with 0·03 milligramme per 10 c.c., but failing with half that amount.

For quantitative purposes a dilute solution should be warmed after the metal has fallen, and after warming hydrochloric acid should be added to bring the precipitate into a state fit for easy filtration; the acid, however, must not be introduced before warming. Silver may be estimated similarly.

Platinum and iridium are not precipitated by alkaline hydrogen peroxide in the cold, so that they can be separated from gold by the above process; ruthenium is attacked, and is therefore not amenable to like treatment. F. H. L.

Separation of Iron and Aluminium by Volatilising the Ferric Chloride. F. A. Gooch and F. S. Havens. (*Zeits. anorg. Chem.*, 1899, xxi., 21.)—When ferric oxide is heated to 180° or 200° C. in a stream of dry hydrochloric acid gas, the bulk of the material (about 90 per cent.) is rapidly converted into chloride and volatilises; the residue is almost black, insoluble in water, slightly soluble in cold (aqueous) hydrochloric acid, but fairly soluble in the hot acid, yielding a solution of ferric chloride. This dark substance, presumably an oxychloride, is only attacked by gaseous hydrochloric acid at the above temperature quite slowly, producing finally a whitish residue composed essentially of ferrous chloride, which is scarcely capable of volatilisation at 500° C. This phenomenon of dissociation can be prevented, and complete volatilisation attained, either by first heating the oxide to 450° or 500° C., and then passing a rapid current of hydrochloric acid vapour, or by carrying out the process at 180° or 200° C. in an atmosphere of hydrochloric acid containing a little free chlorine—the latter method is distinctly to be preferred. Mixtures of ferric oxide with alumina (0·1 to 0·2 gramme of each) can thus, as the examples quoted show, be quantitatively separated at 200° by treatment with the two gases for from half-an-hour to one hour. F. H. L.

Volumetric Estimation of Nickel and Zinc in German Silver and Similar Copper Alloys. J. Jedlicka. (*Listy chemické*, 1899, xxiii., 25; through *Chem. Zeit. Rep.*, 1899, 256.)—One gramme of the finely-powdered alloy is dissolved in the minimum of nitric acid, and evaporated on the water-bath with a little sulphuric acid. The residue is diluted, neutralized, freed from iron with sodium succinate, the copper is reduced with sulphurous acid, thrown down with potassium thiocyanate, and the sulphurous acid driven off from the filtrate. The liquid, which

contains the nickel and zinc (it must not contain any ammonium salts), is heated to boiling in a large porcelain basin, mixed with sodium carbonate solution, and oxidized with sodium hypochlorite, keeping the whole faintly alkaline, so that the nickel may be completely thrown down as peroxide. In order to make the precipitate filter easily, only the smallest necessary quantities of the reagents are added; and to prevent it adhering to the vessel, the parts of the basin uncovered by the liquid are cooled with water before introducing the hypochlorite. The precipitate, consisting of nickel peroxide and basic tin carbonate, is boiled and washed by decantation three times, and filtered through asbestos. It and the asbestos are next rinsed into a beaker, and decomposed by stirring and warming to 40° or 50° C. with the needful volume of semi-normal oxalic acid, which is partly oxidized and partly combines with the nickel and zinc. When the last dark-coloured particles of nickel peroxide have disappeared, the carbon dioxide is driven off by boiling, and the solution is titrated with $\frac{N}{2}$ alkali; it is then diluted sufficiently, acidified with sulphuric acid, and titrated with permanganate containing 10 or 15 grammes of the potassium salt per litre. Small amounts of cobalt (0·3 to 0·5 per cent.) cause a trifling error, but the process only occupies two or three hours, and with care gives results within 0·1 or 0·2 per cent. of the truth. F. H. L.

The Volumetric Estimation of Sulphur in Iron by means of Arsenious Acid. J. Thill. (*Zeit. anal. Chem.*, 1899, xxxviii., 342-344.)—This method is based on the liberation of sulphuretted hydrogen and its subsequent absorption by decinormal arsenious acid solution, 1 c.c. of which is equivalent to 0·002556 gramme of hydrogen sulphide, or 0·0024045 gramme of sulphur.

In making an estimation of the sulphur in iron, steel, etc., the usual method of liberating the sulphur as hydrogen sulphide, and absorbing the gas by a reagent, is followed. Care must be taken, however, that the hydrochloric acid used to dissolve the metal is added as slowly as possible. After the requisite quantity of acid has been added, and the contents of the flask have begun to boil, the flame is so regulated that the boiling only just continues. In this way the distillation of too much hydrochloric acid is prevented.

For the absorption, the author prefers the apparatus devised by E. Franke, but states that the well-known apparatus of Weigert and von Reis can also be used, though with the latter it is advisable to place an additional condensation bulb between the generating and absorption flasks. The latter is charged with 25 c.c. of a decinormal solution of arsenious acid and 50 c.c. of a cold-saturated solution of sodium bicarbonate.

When the iron is completely dissolved the current of carbon dioxide is passed through the apparatus until the whole of the sulphur has been precipitated as arsenious sulphide, which takes place in about ten minutes. The liquid is then rendered acid by means of a few c.c. of hydrochloric acid, made up to 500 c.c., shaken and filtered. The residual arsenious acid is titrated in an aliquot part of the filtrate in the usual manner.

The following are some of the results obtained by this method and by the hydrogen peroxide method:

	Hydrogen Peroxide Method. Sulphur, per cent.		Arsenious Acid Method. Sulphur, per cent.
Thomas pig-iron	0·108	...	0·105
,, ,,	0·081	...	0·083
,, ,,	0·095	...	0·090
,, ,,	0·148	...	0·148

C. A. M.

The Use of Potassium Ferrocyanide for Preparing Standard Ferric Solutions for Volumetric Purposes. K. Schröder. (*Chem. Zeit.*, 1899, xxiii., 533, 540, 557.)—The author finds that potassium ferrocyanide is one of the best substances for preparing an iron solution for volumetric work. It has the advantage over iron filings and wire of being homogeneous, and over solutions (of Mohr's salt, etc.) of being unaffected by changes in temperature. It can be converted into a ferric salt quickly and with little trouble; and the usual destruction or removal of superfluous oxidizing material is avoided. Its purity need only be examined once, determining the proportion of water and that of iron by a gravimetric process; for it keeps indefinitely in brown or black bottles.

From the commercial article a pure product is obtained by rapid recrystallization from a boiling saturated solution, draining the crystals with the pump, drying them for twenty-four hours between paper, then digesting them with absolute alcohol, draining and drying for forty-eight hours as before. For use, 4 or 5 grammes are rinsed into a 500 c.c. flask of Jena glass, with 20 or 25 c.c. of sulphuric acid (specific gravity 1·84); the vessel is placed slantingly on a tripod, and is heated over a bare flame which does not touch the flask, till, after a few agitations, solution is affected. The flame is raised till the carbon monoxide is wholly driven off, then raised further until the liquid boils briskly (using a coil of platinum to prevent bumping). It was found by the author that the iron by this treatment was completely converted into the ferric state, the salt formed being, according to Fownes, ammonium-potassium-iron alum—

$$(NH_4)_2SO_4 + K_2SO_4 + 2Fe_2(SO_4)_3.$$

After thirty minutes it is cooled, 25 c.c. of water are introduced and mixed with the solution; 10 or 15 c.c. of 1.19 hydrochloric acid are next run in, and the whole is boiled for five or ten minutes. The liquid which contains all the iron in the ferric state is ready to be employed as a means of standardizing stannous chloride or (obviously) iodine, if an excess of tin be taken. The titration must not be carried out in flasks with a pronounced yellow tint, or the end-reaction will be difficult of observation. The figures quoted by the author respecting the composition of recrystallized potassium ferrocyanide show a satisfactory agreement with the formula $K_4Fe(CN)_6 + 3H_2O$. F. H. L.

Valuation of Persulphates. G. H. Mondolfo. (*Chem. Zeit.*, 1899, xxiii., 699.)—This process depends on the reaction:

$$M'SO_4 + KI = M'KSO_4 + I.$$

Two or three grammes of the sample are dissolved in 100 c.c. of cold water, 10 c.c. of the liquid are mixed with an excess (0·25 to 0·5 gramme) of potassium

iodide, and warmed for ten minutes to 60° or 80° C. The liberated iodine is titrated with decinormal thiosulphate and starch, 1 c.c. of the reagent corresponding to 0·0114 gramme of NH_4SO_4, or to 0·0135 gramme of KSO_4. F. H. L.

Estimation of Acids from the Volume of Hydrogen they evolve on Treatment with Metals. K. Ulsch. (*Chem. Zeit.*, 1899, xxiii., 624.)—The author has already described (*Zeits. anal. Chem.*, 1891, xxx., 175; 1892, xxxi., 392) a process of determining nitric acid by reducing it to ammonia with nascent hydrogen, and noting the deficit of gas liberated when an excess of iron and a known quantity of sulphuric acid are employed to generate the latter. It has been suggested that occlusion might interfere with the accuracy of the method; further experiments have therefore been instituted which disprove this objection, and show that the process is available for the gas-volumetric estimation of inorganic and many organic acids. To generate the hydrogen, 2·5 grammes of "ferrum pulveratum" and 10 drops of a 5 per cent. solution of platinic chloride are used, but within reasonable limits the exact quantities are immaterial. The volume of gas is read off after a few minutes' shaking, for although a little continues to be evolved for several hours, the reaction is practically completed at once. The strength of the acid may vary within wide limits, but it should not be too weak [apparently it should be about decinormal]. Owing to solubility and the presence of oxygen, the yield of hydrogen is always slightly below the theoretical: 20 c.c. of decinormal sulphuric acid evolve 22·05 c.c. instead of 22·34 c.c. As an example of the accuracy attainable, normal sulphuric acid was prepared by diluting strong acid according to the indications of the above process; 10 c.c. finally neutralized 28·65 c.c. of baryta-water, whereas 10 c.c. of normal acid standardized on sodium carbonate neutralized 28·70 c.c. Hydrochloric acid may be treated similarly, also phosphoric acid, but owing to the formation of ferric phosphate, the latter reaction is slower, and the same portion of iron cannot be used so often. Oxalic, acetic, and succinic acids give correct results; lactic acid (possibly by reason of impurity) comes out too low; tartaric and citric acids present certain difficulties, liberating too much gas. Further details are promised. F. H. L.

Tests for Boric Acid. V. Lenher and J. S. C. Wells. (*Journ. Amer. Chem. Soc.*, vol. xxi. [5], pp. 417-420.)—An improved method of performing the flame-test has been devised by the authors, the apparatus employed being a test-tube containing the substance and reagents, and closed by a cork fitted with a small glass tube drawn out to a fine jet. Heat being applied to the test-tube, and the alcoholic vapour ignited, the flame will show the presence of 0·1 per cent. of boron trioxide, the test thus being ten times as delicate as the ordinary flame-test. Copper and barium salts do not colour this flame.

The authors determined the limits of sensitiveness of the other tests in use, with the following results: Boric ether flame (old method), 1·0 per cent.; glycerin method, 0·1 per cent.; potassium fluoride and acid sulphate, 1·0 per cent turmeric paper, 0·01 per cent. C. S.

Valuation of Hydrogen Peroxide. A. Sonnié-Moret. (*Rép. Pharm.*, 1899, [3], xi., 289; through *Chem. Zeit. Rep.*, 1899, 224.)—Three or four c.c. of the solution are brought into a nitrometer filled with mercury, 1 or 2 c.c. of sodium hydroxide solution are added, and 0·3 or 0·4 gramme of finely powdered manganese dioxide is introduced into the liquid through the mercury by means of a glass tube. The volume of gas is then read off. F. H. L.

The Valuation of Basic Slag (Thomas Meal) on the Basis of its Percentage of Soluble Phosphoric Acid. P. Wagner. (Pamphlet published by Paul Parey, Berlin, 1899.)—So many changes have been made during the past few years in the treatment of Thomas slag, with the object of increasing the solubility of the phosphoric acid, that its composition now differs essentially from that formerly produced. The proportion of easily-decomposable calcium silicate now present is much greater than was formerly the case, with the consequence that the combined action of this and the free lime present is sufficient to so reduce the acidity of the official (German) citrate solution, as to affect its solvent action upon the phosphoric acid of the slag.

The author has carried out a series of experiments with the view of determining the relative manurial values of the slags now on the market, and of so modifying the official method as to adapt it to suit the altered circumstances. He finds that a citrate solution containing 2 per cent. of free citric acid is preferable to the official solution containing only 1·4 per cent. For testing pure slags, a simple aqueous citric acid solution may be used, but the acidified citrate solution has this advantage, that it affords a means of distinguishing between pure slags and those mixed with ground Redonda or Belgian phosphates. Thus, a Belgian phosphate treated with a citrate solution containing 2 per cent. of free citric acid showed a solubility of only 8 per cent., whilst the same phosphate treated with a 2 per cent. aqueous citric acid solution showed a solubility of 30 per cent. The following are abridged directions of the process recommended by the author:

I. Solutions:

1. A 10 per cent. citric acid stock solution containing 5 grammes of salicylic acid per 10 litres.

2. The same diluted to a 2 per cent. strength.

3. Molybdate solution: 150 grammes of ammonium molybdate to be dissolved in 500 c.c. of water, 1 litre of nitric acid of 1·19 specific gravity, and 400 grammes of ammonium nitrate to be added, and the whole diluted with water to 2 litres. The solution to be allowed to stand for twenty-four hours at 35° C., and then filtered.

4. Magnesia mixture: 110 grammes of magnesium chloride and 140 grammes of ammonium chloride to be dissolved in 1,300 c.c. of water, and 700 c.c. of 8 per cent. ammonia added. The solution to be allowed to stand for several days, and then filtered.

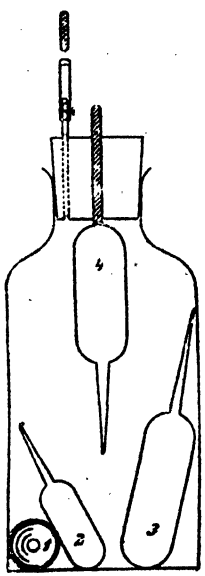
5. Citrate-magnesia mixture: 200 grammes of citric acid to be dissolved in a 20 per cent. ammonia solution, and made up to a litre with the same solution. This solution to be then mixed with 1 litre of the magnesia mixture.

II. The determination is carried out as follows: 5 grammes of the Thomas

meal are placed in a half-litre flask containing 5 c.c. of alcohol, and the flask filled to the mark with dilute (2 per cent.) citric acid solution, at a temperature of 17½° C. The flask is then closed with a caoutchouc stopper, and shaken for thirty minutes in a rotating apparatus making thirty to forty revolutions per minute. The liquid is then filtered. Fifty c.c. of the filtrate are placed in a beaker, 80 to 100 c.c. molybdate solution added, the mixture warmed to 60° to 70° C., and allowed to cool. It is then filtered, the precipitate washed with 1 per cent. nitric acid solution, and dissolved in about 100 c.c. of 2 per cent. ammonia. Fifteen c.c. magnesia mixture are then added, the mixture allowed to stand for two hours, the precipitate filtered off, washed with 2 per cent. ammonia solution, dried, ignited, and weighed.

H. H. B. S.

Determination of Oxygen in Water. L. Mutschler. (*Zeit. für Untersuch. der Nahr. und Genussmittel*, ii., 481-484.)—By means of the apparatus shown in the accompanying figure, the author adapts Mohr's method, for the determination of oxygen in water in situations where the accommodation of a properly-equipped laboratory is not available. The apparatus consists of a strong bottle of about 1 litre capacity with a wide neck, to which is fitted a double-bored caoutchouc stopper. The tubes 2, 3, and 4 are made of thin glass, so that they can be easily fractured by means of the glass or agate ball 1. They are filled with the reagents and sealed up. The tube 4 is for the sulphuric acid. It is of 15 to 20 c.c. capacity, and is fixed through the central hole in the stopper, as shown in the figure. The tube 2, of 5 to 8 c.c. capacity, is for the soda solution, and the tube 3, of 25 to 30 c.c. capacity, for a known quantity of ferrous ammonium sulphate solution. Through the second hole in the stopper a narrow-bore glass tube is fixed, flush with the under surface of the stopper, and projecting about 2 cm. above the top. On the projecting part is fixed a piece of caoutchouc tubing, which can be closed by means of a small piece of glass rod, thus providing a vent.



The determination is carried out as follows :

The bottle is filled with the water to be tested, the tubes are inserted, the stopper fixed in its place, and the vent-tube closed. The bottle is then carefully shaken so as to fracture and release the contents of the ferrous salt and soda tubes without breaking the sulphuric acid tube. The latter is left intact until after the return to the laboratory, when its contents are similarly discharged. The bottle is then allowed to stand for 10 to 15 minutes until the fluid clears, after which it is washed out with boiled water, and the unoxidized ferrous salt determined by titration.

H. H. B. S.

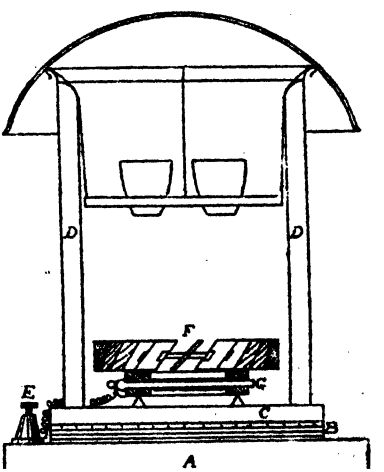
Action of a Hard Water on Certain Metals. J. L. Howe and J. L. Morrison. (*Journ. Amer. Chem. Soc.*, vol. xxi. [5], pp. 422-425.)—The water examined was found

to corrode brass fittings; it contained per 100,000 parts: Lime, 7·80, magnesia, 4·065, carbon dioxide, 30·196, sulphur trioxide, 0·2127 parts, chlorine a trace. Strips of brass, zinc, copper, iron, lead, and aluminium were immersed in the water during four months, with and without access of air, and duplicate tests were made with distilled water. The results of the subsequent examination of the metal and water lead to the conclusion that waters of the above class, free from chlorine, attack zinc more readily than distilled water does, and dissolve out this metal from brass; they should therefore not be conveyed through zinc-lined iron pipes. The hardness of this water does not prevent it dissolving lead, the action being more than half as great as that of distilled water when protected from the air. When air is excluded, the action on iron is relatively slight, and this water has no influence on aluminium or nickel.

C. S.

APPARATUS.

An Electrically-heated Drying Oven. T. W. Richards. (*Amer. Chem. Jour.*, 1899, xxii., 45.)—The general construction of this oven is shown by the annexed drawing. G is a frame composed of two parallel porcelain clamps, such as are used for securing electric wires, held together by means of glass rods passing through the screw-holes, and with larger glass tubes encircling the rods to act as distance pieces. Round the porcelain sides of the frame is wound a coil of platinum wire to act as the resistance. F is a stationary fan made by cutting radial slits in a circle of sheet tin or platinum, bending the sectors through an angle of 45°; or it may be constructed of mica vanes and a platinum centre, etc. Its object is to force the current of hot air to ascend in a spiral in order to produce a uniform temperature in all parts of the oven; this it does to within 2°. The walls, D, consist of two beakers with their bottoms cut off, the annular space being packed with asbestos, etc. The cover is either a clock-glass, a porcelain basin, or a tile. C is a porcelain tile, B layers of asbestos, A a wooden base. The quantity of wire on the resistance must obviously vary according to circumstances. Richards quotes for a 52-volt current, and prescribes 5 metres of No. 32 B. and S. [about No. 35½ S.W.G. = 0·008 inch, or 0·2 millimetre] platinum wire. This when heated has a resistance of about 25 ohms, requires, therefore, 2 ampères (which can safely be taken from a lighting circuit), and absorbs 100 watts—an amount of energy that is capable of heating an oven large enough for most analytical laboratories. If the current be obtained from 8-volt accumulators, from 5 to 15 ampères should be needed, according to the dimensions of the apparatus; in this case thin platinum foil would make the best resistance. The fixed resistance is adjusted to give the maximum temperature; lower heats are



produced by including other resistances outside, or by increasing the amount of air admitted.

An ordinary 16 candle-power glow-lamp fixed inside a paraffin-lamp chimney, the whole jacketed with a tin can, forms an efficient makeshift which will hold a crucible; it gives a temperature of 120° C. or 130° C. F. H. L.

The Incandescent Electric Lamp as a Source of Heat in Ether Extraction. C. G. Hopkins. (*Journ. Amer. Chem. Soc.*, vol. xxi. [8], pp. 645-647.)—For heating a battery of twenty Soxhlet apparatus, the author employs five ordinary 110-volt thirty-two candle-power lamps placed in an air-bath of galvanized iron, 36 × 3 × 6 inches, lagged with asbestos to prevent radiation, the removable cover being perforated by twenty 2-inch holes in two rows. The lamps are set 7 inches apart, under the centres of the squares formed by every four holes, thus equalizing the heat, which is sufficient to distil about 60 drops per minute in each apparatus. The usual temperature of the bath does not exceed 75° C., but since, when all the holes are covered, the heat may attain the ignition point of ether, it is advisable, for absolute security in case of breakage of any of the flasks, to immerse the lamp-bulbs in water. The economy of these lamps is high, about 96 per cent. of the electrical energy being converted into heat. C. S.

REVIEW.

A Treatise on Practical Chemistry and Qualitative Analysis. By Frank Clowes, D.Sc., Emeritus Professor in the University College, Nottingham; Chemical Adviser to the London County Council, etc. Seventh edition. London: Churchill, 1899.

This work, which has now reached its seventh edition, is so well known to students and teachers of chemistry, that any special review of it is almost unnecessary, and criticism is disarmed. It has, in fact, become a standard work, and may be regarded as one of the leading English text-books of Qualitative Analysis.

It has perhaps ever been a vexed question whether detailed directions to the student are on the whole better than a brief outline of the tests to be made by him, but the unqualified success of Dr. Clowes's work may be taken as a strong argument in favour of the former view; and always provided that the student can be induced to read, there can be no doubt that a conscientious study of this manual will insure a sound knowledge of the subject, while the time of the teacher will be largely saved.

The tables of differences constitute a very special feature, and in this and the later editions there is scarcely any branch of the subject which has not been more or less fully handled, notably many organic substances of importance, and the reactions of the rare metals.

There is perhaps only one regrettable omission—that of the theoretical explanations of analytical processes; these I venture to think might still find a place in a new edition. Dr. Clowes is not unconscious of this omission, and alludes to it in his preface. H. W. H.

THE ANALYST.

DECEMBER, 1899.

THE SALE OF FOOD AND DRUGS ACT OF 1899.

[62 & 63 Vic., Ch. 51.]

ARRANGEMENT OF SECTIONS.

Section

1. Precautions against importation of agricultural and other produce insufficiently marked.
2. Power for Local Government Board or Board of Agriculture to sample articles of food.
3. Power for Local Government Board or Board of Agriculture to act in default of local authority.
4. Power for Board of Agriculture to make regulations as to analysis of milk, cream, butter, or cheese.
5. Extension of Margarine Act, 1887, to margarine-cheese.
6. Marking of margarine and margarine-cheese.
7. Provisions as to manufacturers of and dealers in margarine and margarine-cheese.
8. Restriction on amount of butter fat in margarine.
9. Provision as to name and address of person selling milk or cream in a public place.
10. Division of samples taken in course of delivery or transit.
11. Provisions as to condensed, separated, or skimmed milk.
12. Notice of mixtures.
13. Amendment of 38 & 39 Vic. c. 63, as to samples.
14. Taking samples in course of delivery.
15. Amendment of 38 & 39 Vic. c. 63, as to registered parcels.
16. Obstruction of officer in discharge of his duties.
17. Penalties for offences under the Sale of Food and Drugs Acts.
18. Articles sold in tins or packets.
19. Time for proceeding and regulation as to summons.
20. Provisions as to use of warranty or invoice as defence, and proceedings against the warrantor.
21. Duty of court to send articles for analysis.
22. Provisions as to certificates of analysis.
23. Transfer of powers from Secretary for Scotland to Local Government Board.
24. Application to Ireland.

Section
25. Interpretation of terms.
26. Definition of "food."
27. Repeal of enactments in schedule.
28. Short title and commencement.
Schedule.

CHAPTER 51.

AN ACT TO AMEND THE LAW RELATING TO THE SALE OF FOOD AND DRUGS.
[August 9, 1899.]

BE it enacted by the Queen's most Excellent Majesty, by and with the advice and consent of the Lords Spiritual and Temporal, and Commons, in this present Parliament assembled, and by the authority of the same, as follows:

1.—If there is imported into the United Kingdom any of the following articles, namely:

(*a*) Margarine or margarine-cheese, except in packages conspicuously marked "Margarine" or "Margarine-cheese," as the case may require; or

(*b*) Adulterated or impoverished butter (other than margarine), or adulterated or impoverished milk or cream, except in packages or cans conspicuously marked with a name or description indicating that the butter or milk or cream has been so treated; or

(*c*) Condensed separated or skimmed milk, except in tins or other receptacles which bear a label whereon the words "Machine-skimmed Milk" or "Skimmed Milk," as the case may require, are printed in large and legible type; or

(*d*) Any adulterated or impoverished article of food to which Her Majesty may by Order in Council direct that this section shall be applied, unless the same be imported in packages or receptacles conspicuously marked with a name or description indicating that the article has been so treated;

the importer shall be liable, on summary conviction, for the first offence to a fine not exceeding twenty pounds, for the second offence to a fine not exceeding fifty pounds, and for any subsequent offence to a fine not exceeding one hundred pounds.

(2) The word "importer" shall include any person who, whether as owner, consignor, or consignee, agent, or broker, is in possession of, or in anywise entitled to the custody or control of, the article; prosecutions for offences under this section shall be undertaken by the Commissioners of Customs; and subject to the provisions of this Act this section shall have effect as if it were part of the Customs Consolidation Act, 1876.

(3) The Commissioners of Customs shall, in accordance with directions given by the Treasury after consultation with the Board of Agriculture, take such samples of consignments of imported articles of food as may be necessary for the enforcement of the foregoing provisions of this section.

(4) Where the Commissioners of Customs take a sample of any consignment in pursuance of such directions, they shall divide it into not less than three parts, and

send one part to the importer and one part to the principal chemist of the Government laboratories, and retain one part.

(5) In any proceeding under this section the certificate of the principal chemist of the result of the analysis shall be sufficient evidence of the facts therein stated, unless the defendant require that the person who made the analysis be called as a witness.

(6) If, in any case, the Commissioners of Customs are of opinion that an offence against this section has been committed, they shall communicate to the Board of Agriculture for their information the name of the importer and such other facts as they possess or may obtain as to the destination of the consignment.

(7) For the purposes of this section an article of food shall be deemed to be adulterated or impoverished if it has been mixed with any other substance, or if any part of it has been abstracted so as in either case to affect injuriously its quality, substance, or nature.

Provided that an article of food shall not be deemed to be adulterated by reason only of the addition of any preservative or colouring matter of such a nature and in such quantity as not to render the article injurious to health.

2.—(1) The Local Government Board may, in relation to any matter appearing to that Board to affect the general interest of the consumer, and the Board of Agriculture may, in relation to any matter appearing to that Board to affect the general interests of agriculture in the United Kingdom, direct an officer of the Board to procure for analysis samples of any article of food, and thereupon the officer shall have all the powers of procuring samples conferred by the Sale of Food and Drugs Acts, and those Acts shall apply as if the officer were an officer authorized to procure samples under the Sale of Food and Drugs Act, 1875, except that—

(*a*) The officer procuring the sample shall divide the same into four parts, and shall deal with three of such parts in the manner directed by section fourteen of the Sale of Food and Drugs Act, 1875, as amended by this Act, and shall send the fourth part to the Board, and

(*b*) The fee for analysis shall be payable to the analyst by the local authority of the place where the sample is procured.

(2) The Board shall communicate the result of the analysis of any such sample to the local authority, and thereupon there shall be the like duty and power on the part of the local authority to cause proceedings to be taken as if the local authority had caused the analysis to be made.

3.—(1) It shall be the duty of every local authority entrusted with the execution of the laws relating to the sale of food and drugs to appoint a public analyst, and put in force from time to time, as occasion may arise, the powers with which they are invested, so as to provide proper securities for the sale of food and drugs in a pure and genuine condition, and in particular to direct their officers to take samples for analysis.

(2) If the Local Government Board or Board of Agriculture, after communication with a local authority, are of opinion that the local authority have failed to execute or enforce any of the provisions of the Sale of Food and Drugs Acts in relation to any article of food, and that their failure affects the general interest of the consumer or

the general interests of agriculture in the United Kingdom, as the case may be, the Board concerned may, by order, empower an officer of the Board to execute and enforce those provisions, or to procure the execution and enforcement thereof in relation to any article of food mentioned in the order.

(3) The expenses incurred by the Board or their officer under any such order shall be treated as expenses incurred by the local authority in the execution of the said Acts, and shall be paid by the local authority to the Board on demand, and in default the Board may recover the amount of the expenses with costs from the local authority.

(4) For the purposes of this section an order of the Board shall be conclusive in respect of any default, amount of expenses, or other matter therein stated or appearing.

(5) Any public analyst appointed under the Sale of Food and Drugs Acts shall furnish such proof of competency as may from time to time be required by regulation framed by the Local Government Board.

4.—(1) The Board of Agriculture may, after such inquiry as they deem necessary, make regulations for determining what deficiency in any of the normal constituents of genuine milk, cream, butter, or cheese, or what addition of extraneous matter or proportion of water, in any sample of milk (including condensed milk), cream, butter, or cheese, shall for the purposes of the Sale of Food and Drugs Acts raise a presumption, until the contrary is proved, that the milk, cream, butter, or cheese is not genuine or is injurious to health, and an analyst shall have regard to such regulations in certifying the result of an analysis under those Acts.

(2) Any regulations made under this section shall be notified in the London and Edinburgh Gazettes, and shall also be made known in such other manner as the Board of Agriculture may direct.

5.—The provisions of the Margarine Act, 1887, as amended by this Act, shall extend to margarine-cheese, and shall apply accordingly, with the substitution of "margarine-cheese" and "cheese" for "margarine" and "butter," and provided that all margarine-cheese sold or dealt in otherwise than by retail shall either be inclosed in packages marked in accordance with the Margarine Act, 1887, as amended by this Act, or be itself conspicuously branded with the words "margarine-cheese."

6.—(1) Where under this Act or the Margarine Act, 1887, it is required that any package containing margarine or margarine-cheese shall be branded or marked, the brand or mark shall be on the package itself, and not solely on a label, ticket, or other thing attached thereto.

(2) The letters required to be printed on the paper wrapper in which margarine or margarine-cheese is sold shall be capital block letters not less than half an inch long and distinctly legible, and no other printed matter shall appear on the wrapper.

(3) The words "or with" in section six of the Margarine Act, 1887, shall be repealed.

7.—(1) Every occupier of a manufactory of margarine or margarine-cheese, and every wholesale dealer in such substances, shall keep a register showing the quantity and destination of each consignment of such substances sent out from his manufactory or place of business, and this register shall be open to the inspection of any officer of the Board of Agriculture.

(2) Any officer of the Board of Agriculture shall have power to enter at all reasonable times any manufactory of margarine or margarine-cheese, and to inspect any process of manufacture therein, and to take samples for analysis.

(3) If any such occupier or dealer—

(*a*) Fails to keep such a register, or

(*b*) Refuses to produce the register when required to do so by an officer of the Board of Agriculture, or

(*c*) Fails to keep the register posted up to date, or

(*d*) Wilfully makes any entry in the register which is false in any particular, or

(*e*) Fraudulently omits to enter any particular which ought to be entered in the register,

he shall be liable on summary conviction for the first offence to a fine not exceeding ten pounds, and for any subsequent offence to a fine not exceeding fifty pounds.

(4) The provisions of section nine of the Margarine Act, 1887, relating to registration of manufactories shall extend to any premises wherein the business of a wholesale dealer in margarine or margarine-cheese is carried on.

(5) The registration of a manufactory or other premises shall be forthwith notified by the local authority to the Board of Agriculture.

8. It shall be unlawful to manufacture, sell, expose for sale, or import any margarine the fat of which contains more than ten per cent. of butter fat, and every person who manufactures, sells, exposes for sale, or imports any margarine which contains more than that percentage shall be guilty of an offence under the Margarine Act, 1887; and any defence which would be a defence under section seven of that Act shall be a defence under this section, and the provisions of the former section shall apply accordingly.

Provided that nothing in this section shall apply to any margarine manufactured or imported in fulfilment of any contract made before the twentieth day of July one thousand eight hundred and ninety-nine.

9. Every person who, himself or by his servant, in any highway or place of public resort sells milk or cream from a vehicle or from a can or other receptacle shall have conspicuously inscribed on the vehicle or receptacle his name and address, and in default shall be liable on summary conviction to a fine not exceeding two pounds.

10. In the case of a sample taken of milk in course of delivery, or of margarine or margarine-cheese forwarded by a public conveyance, the person taking the sample shall forward by registered parcel or otherwise a portion of the sample marked, and sealed, or fastened up, to the consignor if his name and address appear on the can or package containing the article sampled.

11. Every tin or other receptacle containing condensed, separated, or skimmed milk must bear a label clearly visible to the purchaser on which the words "Machine-skimmed Milk," or "Skimmed Milk," as the case may require, are printed in large and legible type; and if any person sells or exposes or offers for sale condensed, separated, or skimmed milk in contravention of this section, he shall be liable on summary conviction to a fine not exceeding ten pounds.

12. The label referred to section eight of the Sale of Food and Drugs Act, 1875,

shall not be deemed to be distinctly and legibly written or printed within the meaning of that section unless it is so written or printed that the notice of mixture given by the label is not obscured by other matter on the label: Provided that nothing in this enactment shall hinder or affect the use of any registered trade mark, or of any label which has been continuously in use for at least seven years before the commencement of this Act; but the Comptroller-General of Patents, Designs, and Trade Marks shall not register any trade mark purporting to describe a mixture unless it complies with the requirements of this enactment.

13. In section fourteen of the Sale of Food and Drugs Act, 1875, the words "offer to" and the words "proceed accordingly and shall" shall be repealed.

14. The provisions of section three and section four of the Sale of Food and Drugs Act Amendment Act, 1879 (relating to the taking of samples of milk in course of delivery), shall apply to every other article of food: Provided that no samples shall be taken under this section except upon the request or with the consent of the purchaser or consignee.

15. In section sixteen of the Sale of Food and Drugs Act, 1875, the words "registered parcel" shall be substituted for the words "registered letter."

16. Any person who wilfully obstructs or impedes any inspector or other officer in the course of his duties under the Sale of Food and Drugs Acts, or by any gratuity, bribe, promise, or other inducement prevents, or attempts to prevent, the due execution by such inspector or officer of his duty under those Acts, shall be liable, on summary conviction, for the first offence to a fine not exceeding twenty pounds, for the second offence to a fine not exceeding fifty pounds, and for any subsequent offence to a fine not exceeding one hundred pounds.

17.—(1) Where, under any provision of the Sale of Food and Drugs Act, 1875, a person guilty of an offence is liable to a fine which may extend to twenty pounds as a maximum, he shall be liable for a second offence under the same provision to a fine not exceeding fifty pounds, and for any subsequent offence to a fine not exceeding one hundred pounds.

(2) Where, under any provision of the Sale of Food and Drugs Acts, a person guilty of an offence is liable to a fine exceeding fifty pounds, and the offence, in the opinion of the court, was committed by the personal act, default, or culpable negligence of the person accused, that person shall be liable (if the court is of opinion that a fine will not meet the circumstances of the case) to imprisonment, with or without hard labour, for a period not exceeding three months.

18. Notwithstanding anything in section seventeen of the Sale of Food and Drugs Act, 1875, where any article of food or drug is exposed for sale in an unopened tin or packet duly labelled, no person shall be required to sell it except in the unopened tin or packet in which it is contained.

19.—(1) When any article of food or drug has been purchased from any person for test purposes, any prosecution under the Sale of Food and Drugs Acts in respect of the Sale thereof, notwithstanding anything contained in section twenty of the Sale of Food and Drugs Act, 1875, shall not be instituted after the expiration of twenty-eight days from the time of the purchase.

(2) In any prosecution under the Sale of Food and Drugs Acts the summons

shall state particulars of the offence or offences alleged, and also the name of the prosecutor, and shall not be made returnable in less time than fourteen days from the day on which it is served, and there must be served therewith a copy of any analyst's certificate obtained on behalf of the prosecutor.

20.—(1) A warranty or invoice shall not be available as a defence to any proceeding under the Sale of Food and Drugs Acts unless the defendant has, within seven days after service of the summons, sent to the purchaser a copy of such warranty or invoice with a written notice stating that he intends to rely on the warranty or invoice, and specifying the name and address of the person from whom he received it, and has also sent a like notice of his intention to such person.

(2) The person by whom such warranty or invoice is alleged to have been given shall be entitled to appear at the hearing and to give evidence, and the court may, if it thinks fit, adjourn the hearing to enable him to do so.

(3) A warranty or invoice given by a person resident outside the United Kingdom shall not be available as a defence to any proceeding under the Sale of Food and Drugs Acts, unless the defendant proves that he had taken reasonable steps to ascertain and did in fact believe in the accuracy of the statement contained in the warranty or invoice.

(4) Where the defendant is a servant of the person who purchased the article under a warranty or invoice he shall, subject to the provisions of this section, be entitled to rely on section twenty-five of the Sale of Food and Drugs Act, 1875, and section seven of the Margarine Act, 1887, in the same way as his employer or master would have been entitled to do if he had been the defendant, provided that the servant further proves that he had no reason to believe that the article was otherwise than that demanded by the prosecutor.

(5) Where the defendant in a prosecution under the Sale of Food and Drugs Acts has been discharged under the provisions of section twenty-five of the Sale of Food and Drugs Act, 1875, as amended by this Act, any proceedings under the Sale of Food and Drugs Acts for giving the warranty relied on by the defendant in such prosecution, may be taken as well before a court having jurisdiction in the place where the article of food or drug to which the warranty relates was purchased for analysis as before a court having jurisdiction in the place where the warranty was given.

(6) Every person who, in respect of an article of food or drug sold by him as principal or agent, gives to the purchaser a false warranty in writing, shall be liable, on summary conviction, for the first offence to a fine not exceeding twenty pounds, for the second offence to a fine not exceeding fifty pounds, and for any subsequent offence to a fine not exceeding one hundred pounds, unless he proves to the satisfaction of the court that when he gave the warranty he had reason to believe that the statements or descriptions contained therein were true.

21. The justices or court referred to in section twenty-two of the Sale of Food and Drugs Act, 1875, shall on the request of either party under that section cause an article of food or drug to be sent to the Commissioners of Inland Revenue for analysis, and may, if they think fit, do so without any such request.

22.—(1) At the hearing of the information in any proceeding under the Sale of

Food and Drugs Acts, the production by the defendant of a certificate of analysis by a public analyst in the form prescribed in section eighteen of the Sale of Food and Drugs Act, 1875, shall be sufficient evidence of the facts therein stated, unless the prosecutor requires that the analyst be called as a witness.

(2) A copy of every such certificate shall be sent to the prosecutor at least three clear days before the return day, and if it be not so sent the court may, if it thinks fit, adjourn the hearing on such terms as may seem proper.

23. This Act shall apply to Scotland with the substitution for "the Local Government Board" of "the Local Government Board for Scotland," and all powers and duties vested in or imposed on the Secretary for Scotland in relation to the Sale of Food and Drugs Acts shall be transferred to, vested in, or imposed on the Local Government Board for Scotland.

24. This Act shall apply to Ireland with the substitution for "the Board of Agriculture" of "the Department of Agriculture and Technical Instruction for Ireland," and for "the Local Government Board" of "the Local Government Board for Ireland," and for "the London and Edinburgh Gazettes" of "the Dublin Gazette."

25. In this Act, unless the context otherwise requires—

The expression "margarine-cheese" means any substance, whether compound or otherwise, which is prepared in imitation of cheese, and which contains fat not derived from milk:

The expression "cheese" means the substance usually known as cheese, containing no fat derived otherwise than from milk:

The expression "local authority" means any local authority authorized to appoint an analyst for the purposes of the Sale of Food and Drugs Acts, and the expression "public analyst" means an analyst so appointed:

Other expressions have the same meaning as in the Sale of Food and Drugs Acts, and an offence under this Act shall be treated as an offence under those Acts.

26. For the purposes of the Sale of Food and Drugs Acts the expression "food" shall include every article used for food or drink by man, other than drugs or water, and any article which ordinarily enters into or is used in the composition or preparation of human food; and shall also include flavouring matters and condiments.

27. The enactments in the schedule to this Act are hereby repealed to the extent mentioned in the third column of that schedule.

28.—(1) This Act may be cited as the Sale of Food and Drugs Act, 1899, and the Sale of Food and Drugs Act, 1875, and the Sale of Food and Drugs Act Amendment Act, 1879, and the Margarine Act, 1887, and this Act may be cited collectively as the Sale of Food and Drugs Acts, 1875 to 1899, and are in this Act referred to as the Sale of Food and Drugs Acts.

(2) This Act shall come into operation on the first day of January one thousand nine hundred.

SCHEDULE.

ENACTMENTS REPEALED.

Session and Chapter.	Short Title.	Extent of Repeal.
38 & 39 Vic., c. 63.	The Sale of Food and Drugs Act, 1875.	In section two, the definition of the term "food." In section fourteen, the words "offer to," and the words "proceed accordingly and shall." Section fifteen. In section twenty-seven, the words from "Every person who shall give a false warranty in writing" to "a penalty not exceeding twenty pounds."
42 & 43 Vic., c. 30.	The Sale of Food and Drugs Act Amendment Act, 1879.	Section ten.
50 & 51 Vic., c. 29.	The Margarine Act, 1887.	In section six, the words "or with" and the words "not less than a quarter of an inch square."
54 & 55 Vic., c. 46.	The Post-Office Act, 1891.	Section eleven.

PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS.

THE monthly meeting of the Society was held on Wednesday evening, November 1, in the Chemical Society's Rooms, Burlington House, the President (Mr. W. W. Fisher, M.A.) occupying the chair.

The minutes of the previous meeting were read and confirmed.

Certificates of proposal for election to membership in favour of the following candidates were read for the second time: E. C. P. Barber, Chemist to the Anglo-Chilian Nitrate Railway Company, Toco, Chili; Frederick Davis, 51, Imperial Buildings, Ludgate Circus, London; William Francis, Chief Analyst to the Condensed Milk Company, of Ireland, Limerick; Alfred Lucas, Analytical Chemist, Public Works Department (Geological Survey Branch), Cairo; Edward Russell, B.Sc. (Lond.), F.I.C., Demonstrator of State Medicine, King's College, London; and F. L. Slocum, Ph.D., Pittsburgh, Pa., U.S.A. And in favour of the following candidates for the first time: John Alfred Foster, A.I.C., Assistant Chemist, Her Majesty's Dockyard, Portsmouth; J. B. P. Harrison, A.I.C., Assistant Analyst to the Aylesbury Dairy Company, London; and D. Lloyd Howard, of Messrs. Howard and Sons, Stratford.

Messrs. T. W. Glass, B.Sc., H. D. Hewitt, D. L. Thomas, and S. A. Woodhead, B.Sc., were elected members of the Society.

The PRESIDENT, referring to the subject of Food and Drugs legislation, said

that since the Society's last meeting the Sale of Food and Drugs Act, 1899, had received the Royal Assent, and would come into operation on January 1 next. It was satisfactory to note that the representations which the Society had made through its officers to the Government while the Bill was being considered had been favourably received, and for the most part adopted in the Act, which showed a considerable improvement when compared with the Bill as first proposed. With regard to the clause which empowered the Board of Agriculture to direct officers to take samples under certain circumstances, there had arisen, from the manner in which the clause was worded in the Bill, an uneasy feeling that it might be interpreted as an attempt to centralize the work of analysis. The Society, however, had been assured by the President of the Board of Agriculture that there was no such intention, and it had been made perfectly clear that such samples would be submitted to the public analyst for the district in which they were taken, and treated as part of his ordinary local work. The central idea which the Society had expressed in regard to any further legislation—viz., that something in the nature of a standing departmental committee of reference should be established—was not given effect to in the form in which it was suggested, but a nucleus had been established by the new Act, and it remained to be seen in the future how far it would be developed and made useful. The wording of the section relating to the proportion of butter-fat to be permitted in margarine has been modified in accordance with a suggestion made by the Council, and a proposal which the draft Bill contained, involving an alteration of the form of certificate, had been abandoned, the form of certificate remaining as it was. The provision that any legal proceedings must be taken within twenty-eight days of the time of purchase would probably not inflict any great hardship on public analysts, given a reasonable amount of consideration on the part of those whose duty it was to purchase and submit samples. Various new articles had been brought within the Act, and one of the most useful alterations was the amendment of the definition of the word "food," the necessity for which had been impressed strongly upon the Government. Altogether, a distinct amount of progress had resulted from the recommendations which the Council had laid before the Government. It might also be mentioned that a committee had been recently appointed by the Local Government Board to inquire into the use of preservatives and colouring matters in food, and had invited the Society's evidence on the subject, in the collection of which members had been asked to assist the Council by recording the results of their experience in a schedule which had been issued to them for the purpose.

A paper was read by Dr. J. Lewkowitsch on "The Meaning of the Acetyl Value in Fat Analysis."



THE MEANING OF THE ACETYL VALUE IN FAT ANALYSIS.

By J. Lewkowitsch, Ph.D.

(*Read at the Meeting, November* 1, 1899.)

The following lines are a continuation of a paper on "The Acetyl Value" which I read before the Society of Chemical Industry (1897, 503). Since that time the processes described have been very extensively used in my laboratory, and their accuracy has been confirmed, not only by different workers in my own laboratory, but also by other chemists. It may, however, be useful to add some explanatory directions.

The preparation of the acetyl product should be done as described previously. A number of experiments have shown that it is quite sufficient to wash the product three times. Prolonged washing beyond the stated limit causes slight dissociation of the acetyl product; of course, this would lead to too low acetyl values. It should be noted that dissociation of the neutral acetyl product takes place in course of time, and the splitting off of the acetyl group may be likened to the natural hydrolysis of the fats and oils. The outward sign of such hydrolysis having taken place is the more or less strong smell of acetic acid which the substance gives off on slight warming. On washing with water the latter acquires a distinct acid reaction. A measure of the amount of hydrolysis will be found in Column VIII. of Table No. I.

The weight of the acetylated product required for the determination of the acetyl value should preferably be increased to about 5 grammes, as for this quantity 1 c.c. of $\frac{N}{10}$ KOH corresponds with about one unit in acetyl value.

The distilled water used in determining the value by either the distillation or filtration process should be carefully freed from CO_2 by previous boiling, as otherwise serious errors may be made. Even the water used for generating steam in the distillation process should be brought to violent ebullition before the steam is passed into the distilling flask. This source of error may easily creep in in the case of very hard water. Check experiments with pure acetic acid will readily guide the operator, if necessary. In order to facilitate the separation of the insoluble fatty acids in the filtration process, it will be found useful to add a slight excess of mineral acid. Of course, this amount must be measured accurately and deducted from the alkali required for determining the dissolved acids.

A glance at the following tables will show that the two processes yield concordant results, especially in the case of those oils which contain but small amounts of volatile fatty acid.* Those fats and oils, however, which are characterized by a large proportion of fatty acids standing on the border line between easily soluble and insoluble fatty acids — such as cocoanut and palm-nut oils — are more conveniently examined by the distillation process; for in this case the fatty acids have a better chance of being well agitated with hot water. It is also necessary to collect at least 600 to 700 c.c. of the distillate (or filtrate, as the case may be) in

* Compare also my paper on the "Theory of Saponification," *Proc. Chem. Soc.*, 1899, 190.

order to obtain the total amount of volatile acids; but even at best it is a somewhat tedious operation to drive off all the fatty acids of cocoanut and palm-nut oils.

I now give in table No. I. a number of determinations made with various fats and oils. In Column V. are registered the acetyl values, as obtained by examining the acetylated products by the two processes described in my first paper. These values I have termed "Apparent Acetyl Values." Owing to the presence of volatile fatty acids, notably in the case of croton oil, palm-nut oil, cocoanut oil, and butter-fat, the apparent acetyl values would be found too high; it is therefore necessary, in order to arrive at the true acetyl value, to determine the amount of volatile fatty acids in the fat or oil before acetylation, and to deduct their amount from the apparent acetyl value. The former values are given in Column II., under the heading, "Total Volatile Fatty Acids per gramme, in terms of mgrms. KOH." They have been determined mostly by the distillation process, in some cases by the filtration process as well, in order to check the results. It is obvious in the case of the four last-mentioned fats that these values cannot be neglected. But even in the remaining cases they are not always negligible, and they even vary for one and the same fat, showing as they do the state of freshness of the samples (compare below). Taking cotton-seed oil as an instance, sample No. I., representing an indifferently refined oil, contains a notable amount of volatile fatty acids when compared with sample VII., the volatile fatty acids of which are almost negligible. (In passing, I may remark how erroneous it would be to draw conclusions from one sample of an oil only.) The difference between the apparent acetyl value and the total volatile fatty acids is given in Column VI., under the heading "True Acetyl Value." I have explained in my first paper (*Jour. Soc. Chem. Ind.* 1897, 504) why the difference of the saponification values of the original and the acetylated fats and oils should not be taken as the true acetyl value; and the same stricture might apply to the differences registered in Column VI. However, as the total volatile fatty acids in the case of castor oil are nil, and in all other cases, with the exception of the last four oils, very small, and, again, as in the last cases the acetyl values are not very high, no appreciable error is being committed.

As regards the difference of saponification values registered in Column VII. in the case of castor oil, which consists almost exclusively of glycerides of hydroxyacids, the incommensurability of the values of the original and acetylated oil becomes very pronounced, and therefore it is no longer permissible to look in this case to the difference as a guide. Yet in almost all other cases enumerated in the following tables this difference may be used as an approximate check of the determination of the acetyl value—in fact, on comparing the values in Columns VI. and VII., the agreement will be found satisfactory, with the exception, of course, of castor oil, as has been explained above. The agreement is satisfactory because the fats examined contained only small quantities of free fatty acids, which give rise to the formation of anhydrides (*cf.* "Proceedings Chemical Society," 1890; 71, 92), and therefore might have interfered with the reliability of the check. In natural fats which have not reached a very advanced state of decomposition (or hydrolysis)—and here we are only concerned with such—the amount of free fatty acids is as a rule small. Hence this question need not be pursued any further.

The acetyl value has been classed as a constant; one would therefore expect the *true* acetyl value to be constant. The table reveals the fact that this is not always the case, the deviations being much greater than the experimental error would warrant. Before discussing the question whether the acetyl value is a constant, or must be regarded as a variable, like the acid value of fats and oils, it will be advisable to inquire as to the substances, or classes of substances, which the acetyl value measures.

From the fact that castor oil, known to consist mostly of glycerides of hydroxylated fatty acids—hydroxyoleic or ricinoleic acid—possesses the highest acetyl value, viz., about 150, whilst the pure triglyceride of ricinoleic acid has the theoretical acetyl value of 159·1, it would appear that the acetyl value is a measure of hydroxylated fatty acids; indeed, originally the acetyl value was proposed to measure them, and has hitherto been regarded a measure thereof.

Alcohols in a fat or oil would, of course, also show an acetyl value; in this case the value is identical with the saponification value of the acetates. The amount of cholesterol and phytosterol in a fat or fatty oil rarely exceeds 1 per cent., hence the presence of these will hardly affect the acetyl value. In fact, as an easy calculation will show, the influence they exercise on the value will be found to be within the limits of the experimental errors of the method. This will be different in the case of waxes, which contain notable amounts of free alcohols.

In order to arrive at definite figures, I have determined the acetyl values of a number of waxes (see Table No. II.).

TABLE No. II.

Kind of Wax.	ORIGINAL WAX.		ACETYLATED WAX.					
	I.	II.	III.	IV.	V.		VI.	VII.
	Saponification Value.	Total Volatile Fatty Acids per Gramme, in terms of Milligrammes KOH.	Saponification Value.	Hehner Value.	Apparent Value. Distillation Process.	Apparent Value. Filtration.	True Acetyl Value.	Difference, III.-I.
Liquid waxes:								
Sperm oil, Northern ...	127·9	2·41	132·6–133·3	99·94	6·95	6·85	4·49	5·05
,, ,, ,, best	132·6	1·36	139·1–140·3	99·94	8·56	7·03	6·43	7·1
,, ,, Southern ...	125·2	1·48	130·9	100·0	7·63	5·83	5·25	5·7
Arctic sperm I.	133·7	1·29	139·5–140·4	100·2	8·00	7·28	6·35	6·25
,, ,, II.	133·2	1·40	137·5	99·99	6·16	4·88	4·12	4·3
Solid waxes:								
Carnauba wax	79·68	2·26	116·0	93·4	57·6	57·4	55·24	36·32
Beeswax	93·5	2·16	107·35	100·2	17·4		15·24	13·85
Wool wax		9·30		98·6	33·7	31·5	23·3	
Spermaceti	122·7	1·97	128·8	102·0	4·7	4·5	2·63	1·1
Wool-wax alcohols ...	0·0	2·2	142·8–143·8		141·2	143·8	140·3	143·3

Speaking broadly, we find the same general outlines as in Table No. I. Whilst it is certain that in the case of carnauba wax, beeswax, and wool wax, the true acetyl values indicate the presence of free alcohols, further research must show whether the same can be said of the other waxes enumerated in the table. The last column shows that also in the case of waxes the difference of the saponification values of the original

and the acetylated substances may be taken as a rough check of the correctness of the direct determination of the true acetyl value, with the exception of carnauba wax, which, on account of the high proportion of free alcohols it is known to contain, affords a very good analogy to castor oil.

TABLE No. III.

	Original Oil.				Acetylated Oil.					
	I.	II.	III.	IV.	V.	VI.	VII.	VIII.	IX.	X.
	Specific Gravity. at 15·5° C.	Saponification Value.	Total Volatile Fatty Acids per Gramme in terms of Milligrammes KOH.	Oxidized Acids Per Cent.	Saponification Value.	Hehner Value.	Apparent Acetyl Value.	True Acetyl Value.	VIII. × ·55	Difference V.-II.
Linseed oil			0·8		205·6	96·2	12·5	11·7	6·4	
Linseed oil, blown two hours at 120° C. ...	0·9334	189·8	1·68	1·2	200·9	94·4	18·9	17·22	9·47	11·1
Linseed oil, blown four hours at 120° C.	0·9403	191·3	3·0	1·7	203·9	94·6	22·5	19·5	10·72	12·6
Linseed oil, blown six hours at 120° C. ...	0·9446	192·4	8·3	5·03	208·2	93·2	25·5	17·2	9·46	15·8
Linseed oil, blown ten hours at 120° C. ..	0·9460	192·7	0·9	7·1	211·8	92·1	32·6	31·7	17·4	19·1
Cotton-seed oil			0·1		200·2	95·7	7·7	7·6	4·18	
Cotton-seed oil, blown two hours at 120° C.	0·9262	194·3	2·88	0·51	203·9	94·8	14·2	11·32	6·23	9·6
Cotton-seed oil, blown four hours at 120° C.	0·9291	194·9	2·44	0·87	212·0	92·9	22·9	20·46	11·25	17·1
Cotton-seed oil, blown six hours at 120° C. ..	0·9350	196·1	4·60	0·94	215·2	91·9	30·0	25·4	13·97	19·1
Cotton-seed oil, blown ten hours at 120° C.	0·9346	196·8	4·16	1·28	218·4	91·4	35·0	30·84	16·96	21·6
Premier jus			0·58		199·6		3·3	2·72	1·49	
Premier jus, blown four hours at 120° C. ...		202·3		0·70	211·6	92·6	18·8			
Oleic acid			0·95		203·7	99·9	3·0	2·05	1·13	9·3
Oleic acid, blown two hours at 120° C. ...	0·9098	204·9	2·9	0·62	211·1	98·5	14·2	11·3	6·21	6·2
Oleic acid, blown four hours at 120° C. ...	0·9121	206·0	3·6	2·6	217·3	99·4	18·2	14·6	8·03	11·3
Oleic acid, blown six hours at 120° C.	0·9123	208·3	4·3	3·5	223·3	97·8	23·9	19·6	10·78	15·0
Oleic acid, blown ten hours at 120° C. ...	0·9238	213·4	2·9	6·0	227·5	97·5	22·3	19·4	10·67	14·1
Blown rape oil, commercial	0·9714	205·6		24·95			62·04	53·14	29·22	
Blown cotton-seed oil, commercial	0·9722	213·7	16·06	26·45			65·6	48·54	26·7	
Boiled oil, commercial, I.		188·7	1·6	6·5	207·4	91·5	25·6	24·0	13·2	18·7
Boiled oil, commercial, II.		186·1	1·1	4·63	199·7	92·8	18·0	16·9	9·29	13·6
Drying oil, prepared with ozone I.			3·2	2·15	208·2	91·5	29·7	26·5	14·57	
Drying oil, prepared with ozone II.			4·2	3·85	211·6	91·7	24·8	20·1	11·06	
Oxidized acids from solidified linseed oil	Acid value 168	199·2		100·0	316·0		130·2		71·6	136·8

The blown oils give high acetyl values, and it therefore appeared desirable to examine them as well. Table No. III. gives the results of the examination of a number of blown oils prepared both on a commercial scale and in the laboratory; to these are added some boiled oils, as also blown oleic acid. Column IV. gives the apparent acetyl values; by deducting from them the amount of volatile fatty acids in the original oils, stated in Column II., we obtain the true acetyl values, registered in Column VII. It has been widely assumed that the blown oils and also boiled oils contain hydroxyacids. Some years ago Fahrion proposed a method for their quantitative determination, based on the insolubility of these acids in petroleum ether. I have, however, shown* that this method is valueless in the case of castor oil fatty acids, and mixtures thereof with non-hydroxylated fatty acids; I have therefore proposed for these insoluble acids the term "oxidized acids" pending further inquiry into their nature. The acetyl values given in Column VIII., Table No. III., further substantiate my objections; for if we were to assume for these hypothetical hydroxyacids the molecular weight of 300—which is, in fact, the molecular weight of hydroxystearic acid, $C_{18}H_{35}(OH)O_2$—it would be possible to calculate their amount. The calculation has been carried out by multiplying the true acetyl values by 0·55. The result is tabulated in Column IX. At the same time, the oxidized fatty acids have been determined quantitatively by Fahrion's method. They are set down in Column IV. With the exception of the commercial blown oils, these numbers greatly deviate from those given in Column IX., although they run parallel to them, as one would naturally expect. It is also remarkable that the difference of the saponification values (given in Column X.), which we have hitherto been able to use as a check, no longer performs the same duty.

In order to gain some further insight into this question, I have examined the oxidized fatty acids of solid oxidized linseed oil, carefully freed from fatty acids soluble in petroleum spirit. The acetyl value of these acids was found to be 130, which on multiplication by 0·55 gives 71·6 per cent. of oxidized acids, whereas theory would postulate 100 per cent. These oxidized acids possessed the acid value 168, and the saponification value 199. We must therefore assume the presence of lactonic substances, a measure of their amount being furnished by the difference 199–168 = 31.

It might be thought for a moment that the acetyl test would permit a distinction to be made between hydroxylated acids and lactones; but it must not be forgotten that true lactones cannot, according to our present views, assimilate acetic acid. Indeed, Geitel† has shown that stearo-lactone does not react with acetyl chloride.

In the case of commercial blown oils, the true acetyl value approximately agrees with the proportion of oxidized acids; hence we may safely conclude that the acetyl value is a measure of the oxidized acids.

But in all the other cases given in Table No. III. we find that the calculated oxidized acids (compare Column IX.) are very much higher than those found direct. What, then, are the substances which lead to the high acetyl values? Are they hydroxylated acids, which are soluble in petroleum spirit, in contradistinction to

* "Chemical Analysis of Oils, Fats, Waxes," etc., Edit. I., p. 157; Edit. II., p. 204.
† *Jour. Prakt. Chemie*, 1888 [37], 84.

the oxidized acids? We can hardly assume that they are polymerized fatty acids, such as we can obtain from castor oil; for we know that polymerization tends to reduce the capacity for assimilating acetyl groups. It is impossible in the present state of our knowledge to pronounce a definite opinion. One naturally looks round for analogies. Thus, pyromeconic acid, to which the somewhat doubtful formula,

$$\begin{matrix} CH\cdot O\cdot CO \\ \dot{C}H\cdot CO\cdot \dot{C}H_2 \end{matrix}$$

is ascribed, yields on boiling with acetyl chloride, acetylpyromeconic acid of the formula $C_5H_3(C_2H_3O)O_3$.

I may further point out that I have shown some years ago* that the mixed fatty acids from sawarri fat yielded an acetyl value of 14, whereas the fat itself has the acetyl value 6·6 (compare Table I.). I was able to show that these fatty acids contain lactonic substances. But as the *fatty acids* were experimented upon, the evidence is not quite conclusive, for reasons given below. Finally, I may state that I found in the crystals obtained by the action of concentrated sulphuric acid on oleic acid, and subsequent dilution with water, a neutral substance whose saponification value on boiling with alcoholic potash agreed fairly well with the number stearo-lactone would demand. Yet with acetic anhydride it yielded a product having an acetyl value and saponification value fairly corresponding with the numbers required for hydroxystearic acid. I am investigating all these points, and in the meantime, without committing myself to any definite opinion, propose to describe them as "unknown acids." Their presence is indicated by the acetyl values set out in Table No. III.

Further research will also be required to determine the oxidized acids in the presence of true hydroxylated acids and "unknown acids." An investigation with this object in view is being carried on. The separation of hydroxylated acids from the oxidized acids is possible by means of the different solubilities of their salts.

So far, then, it appears that an acetyl value indicates the presence of the following classes of substances:

(*a*) Hydroxyacids.
(*b*) Free alcohols.
(*c*) Oxidized fatty acids.
(*d*) "Unknown acids."

If fats contain mono- and di-glycerides, the presence of these would cause the fat to exhibit an acetyl value. And if the moisture of the atmosphere is capable of hydrolysing triglycerides, then it is not only possible, but, indeed, very likely that natural fats and oils which are not perfectly fresh will contain mono- and di-glycerides. I have shown in another paper,† in agreement with Geitel's researches, that mono- and di-glycerides do occur in partially hydrolysed triglycerides. Here it would only be necessary to examine the question whether fats and oils that have undergone *natural* hydrolysis exhibit notable acetyl values due to this cause. The

* *Jour. Soc. Chem. Ind.*, 1890, 842.
† "The Theory of Saponification," *Jour. Soc. Chem. Ind.*, 1898, 1107. Compare also *Proc. Chem. Soc.*, 1899, 190.

numbers given in Table I. supply an answer to some extent. Besides, I have examined several highly decomposed fats and oils containing a considerable proportion of free fatty acids. The latter were removed in the usual way, and the remaining neutral fats examined. They were expected to contain lower glycerides. The results are set out in Table No. IV.

TABLE No. IV.

Neutral Fat of Oil prepared from:	I.	II.	Acetylated Product. III.	IV.	
	Saponification Value.	Hehner Value.	Acetyl Value.	Saponification Value.	Difference. IV.-I.
Maize oil, containing 30 per cent. free fatty acids	188·7	94·34	29·4	210·3	21·6
	188·8	94·37	29·2	211·9	23·1
Cocoanut oil, containing 3·9 per cent. free fatty acids					
(*a*) Soluble in alcohol	262·3	81·78			
(*b*) Insoluble in alcohol	253·8	86·5			
	253·5	85·8			
Cocoanut oil, containing 13·25 per cent. free fatty acids					
(*a*) Soluble in alcohol	259·8	82·58			
	259·7	81·83			
(*b*) Insoluble in alcohol	251·9	86·8			
Palm oil, containing 31·3 per cent. free fatty acids					
(*a*) Soluble in alcohol	189·7	93·05			
	187·8	89·96			
	194·1	93·22			
(*b*) Insoluble in alcohol	193·4	93·66	18·48	210·6	16·7
	194·1	93·80			
Palm oil, containing 23·8 per cent. free fatty acids	197·9	92·55			
(*a*) Soluble in alcohol	186·0	87·4			
	195·7	93·59			
(*b*) Insoluble in alcohol	196·2	94·28	13·61	209·9	13·6
	196·9				
Palm oil, containing 9·67 per cent. free fatty acids	196·5	92·7			
		93·37			
(*a*) Soluble in alcohol	189·5	89·2			
	197·0				
(*b*) Insoluble in alcohol	195·9	94·62	5·4	203·8	7·4
	196·3	94·34			

The samples of maize oil given in Table No. I. have a very much lower acetyl value than the neutral oil prepared from the rancid maize oil, and both the acetyl value and saponification value of the acetylated oil seem to speak in favour of the presence of lower glycerides. Two samples of cocoanut oil, containing 3·9 per cent. and 13·25 per cent. free fatty acids, calculated as oleic acid, respectively, were also freed from the fatty acids, and the neutral oils, presumably containing lower glycerides, were treated with alcohol, in the hope of resolving them into two fractions which might show differences in saponification and Hehner values, as mono- and di-glycerides, being more readily soluble in alcohol than triglycerides, would pass into the alcoholic solution to a greater extent. The results given in the table do not speak against the presence of lower glycerides. Since, however, the triglycerides of the lower fatty acids occurring in cocoanut oil are also to some extent soluble in alcohol, too much weight must not be attached to the cocoanut oil experiment. No

such complication arises in the case of palm oil. Three samples of palm oil, containing 31·3 per cent., 23·8 per cent., and 9·67 per cent. respectively, were treated in a similar fashion. I give the results in Table No. IV. These certainly point to the presence of lower glycerides. Unfortunately, there was not enough of the portions soluble in alcohol left to determine the acetyl values.

A more complete series of experiments will be taken in hand shortly.

The presence of mono- and di-glycerides in natural fats would thus appear to deprive the acetyl value of its rank as a constant. The uncertainty caused thereby can be eliminated by saponifying the fats and determining the acetyl value of the fatty acids. One would naturally conclude that if the acetyl value of the original fat was due to mono- and di-glycerides, the fatty acids would have a considerably lower acetyl value; if not, the acetyl value of the fatty acids should be approximately the same as that of the original fats.

I have therefore determined the acetyl values of some mixed fatty acids prepared from fats whose acetyl values were known. The results can be gathered from Table No. I. In the following table, No. V., I place side by side the acetyl values of the fats and their mixed fatty acids:

TABLE No. V.

			Acetyl Value of		
			Fat or Oil.	Insoluble Fatty Acids.	Difference.
			I.	II.	I.-II.
1.	Cotton-seed oil...	I.	15·8	17·9	−2·1
2.	,, ,, ...	II.	11·7	14·1	−2·4
3.	,, ,, ...	III.	18·0	21·5	−3·5
4.	Tallow ...		4·55	3·3	+1·25
5.	Palm-nut oil ...	I.	9·7	13·4	−3·7
6.	,, ,, ...	II.	7·1	8·0	−0·9
7.		III.	8·36	2·19	+6·17
8.	Cocoanut oil ...	I.	6·9	5·6	+1·3
9.		II.	5·5	0·7	+4·8
10.		III.	7·91	−2·97	+10·88
11.		IV.	9·45	−0·9	+10·35
12.	Butter-fat ...	I.	5·8	0·0	+5·8
13.	,, ...	II.	8·55	16·7	−8·15
14.	Sawarri-fat ...		6·61	14·03	−7·42

At first sight it appears that they prove nothing, for Nos. 7, 9, 10, 11 and 12 would seem to speak in favour of the presence of lower glycerides in the original fat, whilst in Nos. 3, 5, 13, and 14 we notice a considerable increase in the case of the fatty acids. Finally, Nos. 1, 2, 4, 6, 8 would not prove anything, for the difference falls within the limit of experimental error. To explain the apparently capricious results, one appears to be driven to the assumption that in Nos. 7, 9, 10, 11, 12 the original oils and fats contained lower glycerides, whereas in the cases of Nos. 2, 3, 5, 13, 14 the fatty acids have become oxidized in the process of preparation, thus leading to acetyl values, as explained above.

It will be remembered that this very contingency was one of the reasons which induced me to determine the acetyl values in the fats and oils themselves in preference to the fatty acids.

If there is any truth in the above-made assumption, and if we further adopt the view supported by the best evidence that rancidity is due to the oxidation of the fatty acids, setting in subsequently to the natural hydrolysis of triglycerides by the moisture of the atmosphere, it does not appear improbable that the acetyl number might furnish a measure of the rancidity. The latter must not be considered as coterminous with acidity, for whilst the acidity is only a measure of the hydrolysis of the triglycerides, the acetyl value would not only indicate the dissociation of the triglycerides, but, over and above that, the amount of oxidation which has caused the fats to exhibit those properties which we comprise under the term "rancidity."

In order to put this reasoning to the test, a number of fats should be examined both in the fresh state and after exposure to the atmosphere. Only such fats and oils can be chosen as can be considered practically pure triglycerides, *i.e.*, such as have been freed from vegetable and animal tissue—in short, of organic substances the contact with which causes triglycerides to undergo a rapid decomposition, marked by the occurrence of a high proportion of fatty acids. Therefore, such vegetable oils as commercial palm oil, or unrefined seed oils, or olive oil expressed from the fermented pulp, would not be suitable for our purpose, inasmuch as the presence of putrescible or fermentable matter, or ferments themselves (lipases) induces hydrolysis. Nor would unrefined animal fats lend themselves to such an investigation; nor even butter, which contains legitimately organic non-fatty substances, whereas butter-fat would be more appropriate.

Hitherto I have been able to examine a few fats only. After they had been tested in the fresh state, they were exposed for some little time to the atmosphere. The numbers thus obtained are collated in Table No. VI. The differences given in

TABLE No. VI.

Fresh. *After Exposure.*

Original Oil or Fat.		Acetylated Oil or Fat.						Original Oil or Fat.		Acetylated Oil or Fat.						
I	II.	III.	IV.	V.		VI.	VII.	VIII.	IX.	X.	XI.	XII.		XIII.	XIV.	XV.
Saponification Value.	Total Volatile Fatty Acids per Grammo in terms of Milligrammes KOH.	Saponification Value.	Hehner Value.	Apparent Acetyl Value.		True Acetyl Value.	Difference, III.-I.	Saponification Value.	Total Volatile Fatty Acids per Grammo in terms of Milligrammes KOH.	Saponification Value.	Hehner Value.	Apparent Acetyl Value.		True Acetyl Value.	Difference, X.-VIII.	Difference, XIII.-VI.
				Distillation Process.	Filtration Process.							Distillation Process.	Filtration Process.			
	0·1	200·2	95·7		{7·4 / 8·0}	7·6		193·0	2·1	201·8	94·0	8·3	10·1	7·1	8·8	−0·5
198·2			95·5	7·5				194·2	1·5	200·4			9·44	7·94	6·2	
129·0		204·3			146·9		125·1	180·2	1·3	204·6			146·6	144·2	136·4	
186·45	3·6	189·15		4·75		1·15	2·7	192·7	8·7	199·7			19·15	10·45	7·0	+9·3
192·6	0·61			2·86	2·71	1·97		193·1	0·49			5·0	4·8	4·41		+2·44
198·2	1·55	200·9	94·75	6·1	6·1	4·55	4·3	200·0	2·9	210·7	93·2	14·5	12·7	10·7	10·7	+6·15
	0·58	199·8			3·3	2·72		199·3	2·3	206·2	93·6	11·2	8·2	8·9	6·4	+6·18

Column XV. show that the exposed or rancid fats possess higher acetyl numbers than the fats in the fresh state. In the case of cacao butter and curcas oil, at least, the increase in the acetyl value cannot be attributed to the presence of lower glycerides. Yet the number of experiments made is too small to be conclusive evidence in favour of the opinion that the acetyl value might be a better indication of rancidity than the doubtful colour reactions proposed recently. There is all the more reason to consider this an open question, as the results of the examination of the palm-nut and cocoanut oils made at different periods (*cf.* Table No. I.) are somewhat conflicting.

The view that rancid fats contain hydroxyacids has frequently been put forward, and was apparently supported by the high acetyl values given by several chemists for rancid tallow, butter-fat, etc. Since, however, those acetyl values referred to the fatty acids, and, moreover, as they were determined by a method which I have shown to lead to very unreliable results, they cannot be quoted as favouring my opinion. Recently, Gripper* has determined the acetyl values of a number of old rape oils by my method, but the results do not show any regularity, a fact which can only be explained by the assumption that hydrolysis as well as rancidity have each and severally contributed their quota to make up the acetyl value obtained. I expect that examination of a larger number of fats and oils concurrently with that of their mixed fatty acids will throw some light on this subject.

We have thus seen that to the four classes of substances enumerated above, under *a*, *b*, *c*, *d*, we must add :—(*e*) mono- and di-glycerides, (*f*) rancid fats. In the case of most rancid fats it will be difficult to discriminate between classes *e* and *f*, more elaborate methods of analysis being required than we possess at present. Should they be found, then we shall be in a position to discriminate by analytical methods between acid and rancid fats, and dispense with organoleptic methods.

It will be clear that a value which is a measure of so many different classes of substances, as the acetyl value is, cannot be considered a constant, but must be rather looked upon as a variable until we are in a position to differentiate by analytical methods between hydroxyacids, oxidized, and "unknown acids," and to allocate to each of these substances the respective quota which in their totality build up the acetyl value.

Experiments have been taken in hand to elucidate these points ; at present we must, unfortunately, resign ourselves to pronounce a *non liquet*.

The experiments detailed in the paper have been carried out by my assistants, Messrs. E. J. Read and C. D. Robertshaw. I wish to acknowledge here my indebtedness to them, and especially to Mr. Robertshaw, who has also prepared lantern-slides of the tables given.

Discussion.

The President having invited discussion,

Mr. Hehner said that the author's work in differentiating between the various reasons which caused a fatty body to have an acetyl number was very important. The total acetyl value, measuring as it did the hydroxyl, whether it be the hydroxyl

* *Journal Soc. Chem. Ind.*, 1899, p. 342.

in an alcohol or in an acid, was in his opinion but rarely of such value from a practical point of view as to repay the trouble of determining it, castor oil and blown oils being the most noteworthy exceptions. But from the differentiation of the various hydroxyl groups useful information would probably result. Some of the author's figures seemed to indicate that not only the unsaturated acids in a fat might spontaneously oxidize, but that oxidation might also take place in the saturated acids, since a fat, like cocoanut oil, for instance, which contained very little oleic acid had a moderately high acetyl value. It seemed to him a matter of great importance to ascertain if possible whether the unsaturated oil acids, of the constitution of which we were almost entirely ignorant, contained doubly, or perhaps trebly, linked carbon atoms. The difference in the properties of highly unsaturated oils might possibly be explained by different linking, such as he has suggested. Dr. Lewkowitsch had plainly shown that the so-called oxy-acids of Fahrion consisted of at least two classes of substances, one of which consisted clearly of hydroxylated bodies, whilst the others were not hydroxylated.

Mr. ARCHBUTT said that he had compared the acetyl values respectively determined by the original method of Benedikt and by Dr. Lewkowitsch's process, but without finding the labour involved by the latter (owing to the necessity of having to separately estimate and deduct the potash required to neutralize soluble fatty acids) commensurate, from a practical point of view, with the information obtained, and he had not become convinced that the original process did not answer all practical purposes in the technical analysis of oils for the detection of adulteration. In castor oil the acetyl values obtained by both methods were practically identical, but in samples of blown East India rape oil, blown Ravison oil, and blown cotton-seed oil, considerably higher figures were obtained by the new method (unless the soluble fatty acids were allowed for), as shown by the following figures:

Description of Oil.	Acetyl Value of the Insoluble Fatty Acids by Benedikt's Process.	Acetyl Value of the Glycerides by Lewkowitsch's Method, without allowing for the Soluble Fatty Acids present.
Castor oil	149·5	150·2
Castor oil (another sample)	150·8	150·7
Blown East India rape oil	56·8	80·9
Blown Ravison oil	53·7	79·7
Blown cotton-seed oil	69·6	98·3

If the anhydrides formed in Benedikt's process were not hydrolysed again on boiling with water, one would expect the results to be variable, but practically constant results were obtained by Benedikt's process on the blown oils referred to. Therefore, if it were required to use the acetyl value for differentiating between castor oil and blown oils, or for detecting adulteration of the former with the latter, a practically useful result would be obtained more easily by Benedikt's process. In washing the acetylated glycerides obtained from blown oils, there was a considerable tendency to emulsification on boiling with water, whereas there was not the slightest difficulty in boiling and thoroughly washing out the excess of acetic acid from the acetylated fatty

acids. From a scientific point of view the new process was no doubt the more perfect one, but from the purely practical point of view of the analyst it seemed that the original process supplied all that was needed.

Dr. LEWKOWITSCH, in reply, said he thought he might claim that his investigation was not wanting from the practical point of view. In processes connected with the resolution of partly converted fatty acids and fats—such as the products of sulphuric acid saponification—or in the case of castor oil and turkey-red oils, the acetyl value would be found to be of material assistance, and in fact sometimes the only means of unravelling the mysteries in which such matters were often enshrouded. He agreed that it was not at present possible to differentiate between the different classes of substances indicated by the acetyl value, but it was first of all necessary to ascertain what the acetyl actually indicated. Continental literature stated it to indicate the presence of hydroxylated fatty acids or oxy-acids, but acetyl values would be obtained in fats like cocoanut oil, and considerable confusion resulted from such values being reported as indicating the presence of hydroxylated fatty acids. With regard to the "unknown" acids, what he meant and understood was shown in Column IX. of Table III. Multiplying the true acetyl values by 0·55, values were obtained which were higher than the so-called oxidized fatty acids. The question might be asked, Were not such substances merely hydroxylated fatty acids, which were soluble in petroleum spirit? If they were, the matter was finished, but there was no evidence to support this. In what particular way the oxygen was combined he did not think it was possible at present to say. If in a true hydroxylated acid the hydroxyl group were split off, forming water with the hydrogen of the COOH group, there would result a true lactone which should not absorb acetic acid. Stearolactone had been isolated, and was stated not to react, but he had found another substance to which the same formula seemed to apply, but which gave high acetyl values. As a kind of analogy he had mentioned pyromeconic acid, which did not contain a hydroxyl group, but which yielded a well-defined acetylated product. There was a difference between the saturated and the unsaturated fatty acids as regards oxidation —between, for instance, stearic acid and oleic acid. In the case of stearic acid, hydroxylated acids were not obtained by blowing. In the case of oleic acid, the so-called oxidized fatty acids, insoluble in petroleum spirit, were practically very small in quantity, while those calculated from Column VIII. were much higher. The difference unquestionably pointed to the formation of some other bodies which were able to absorb acetic acid, and these, whatever they were, had been termed provisionally "unknown" acids. Most of the so-called acetyl values that were being passed into literature were merely values due to the oxidation or rancidity of the fats, having no quantitative meaning whatever as regards the original fats. In reply to Mr. Archbutt, he had been able to prove substantially that in Benedikt's process the fatty acids were really converted into their anhydrides when boiled with acetic anhydride, and that such anhydrides were not completely hydrolysed by boiling with water. In the case of blown rape oil, the so-called oxidized fatty acids did agree with those calculated from the acetyl value, which led to the conclusion that this class of substances was, amongst other things, indicated by the acetyl value.

ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

FOODS AND DRUGS ANALYSIS.

Assay of Preparations containing Pilocarpine. H. A. D. Jowett. (*The Wellcome Chemical Research Laboratories Report*, No. 6, 1899, 1-7.)—The mixture of amorphous bases obtained from jaborandi or its preparations by any of the usual methods is dissolved in a small quantity of a saturated alcoholic solution of pilocarpine nitrate, the solution slightly acidified by means of a strong alcoholic solution of nitric acid (freshly prepared), and, after the addition of a crystal of pilocarpine nitrate, left for two hours to crystallize. The crystals are filtered off, washed with the saturated solution of pilocarpine nitrate with the aid of a filter-pump, dried and weighed.

In most cases the author states that the crystalline deposit may be assumed to be pilocarpine nitrate, but in very accurate estimations the melting-point and specific rotation of the substance should be determined.

Assuming that pilocarpine is the only impurity, and that Petit and Polonowsky's constants for the nitrate of that base are correct, the percentage of pilocarpine may be calculated from the specific rotation of the mixed nitrates by the formula

$$p = \frac{100}{43{\cdot}7}(x - 38{\cdot}5),$$

in which p represents the required percentage, and x the specific rotation of the mixture.

If the melting-point exceed 174° C., the substance may be regarded as pure pilocarpine nitrate; and if between 167° and 174° C., as containing from 80 to 90 per cent. of that salt. If the melting-point is lower than 167° C., the substance should be recrystallized from a small quantity of hot absolute alcohol, and the deposit weighed and examined as above.

The author shows that pure pilocarpine nitrate obtained from the small-leaved variety of jaborandi (*Maranham jaborandi*) has the same physical characters as that obtained from the true jaborandi, viz., melting-point, 178° C.; $[\alpha]_D + 82{\cdot}9°$; and solubility in water, 1 in 6·4 at 20° C.

A specimen of pure pilocarpine hydrochloride prepared in the usual way from the nitrate, and purified by repeated crystallization, melted at 204°–205° C., and had a specific rotation of $[\alpha]_D = +91{\cdot}74°$.

In examining these salts for medicinal purposes, the following characteristics and tests are recommended as indicating a sufficient degree of purity:

Pilocarpine Nitrate.—White distinct crystals, unaltered on exposure to the air. Soluble in 6 to 7 parts of water at ordinary temperatures in 146 parts of cold 95 per cent. alcohol, fairly soluble in boiling alcohol, nearly insoluble in ether or chloroform. Melting-point, 176° to 178° C. Specific rotation in aqueous solution, +81° to 83°. No residue on ignition. Gives no precipitate on adding to its concentrated aqueous solution ammonium hydroxide, or a solution of sodium or potassium hydroxide. Gives the characteristic tests for nitrates.

Pilocarpine Hydrochloride.—White crystals, which deliquesce in moist air. Soluble in less than its weight of water, and in 10 parts of absolute alcohol; almost insoluble in ether or chloroform. After being dried at 100° C., it melts at 200° to 204° C. Specific rotation in aqueous solution, +90° to 92°. No residue on ignition. On adding ammonium hydroxide to its concentrated aqueous solution there is no precipitate, while potassium or sodium hydroxide only precipitates a few oily drops, which soon redissolve, and it gives the tests for chlorides. C. A. M.

ORGANIC ANALYSIS.

Chlorine Compounds in Cotton Oil, and its Behaviour with the Phloroglucinol and Vanillin Test. P. N. Raikow. (*Chem. Zeit.*, 1899, xxiii., 769, 802.)—The fact that cotton oil gives a red colour with the author's phloroglucinol and vanillin test (ANALYST, 1898, xxiii., 244) has hitherto been explained, in accordance with Dupont's definite assertions, by the statement that the material contains an organic compound of sulphur. After examining four samples of cotton oil, however, obtained from Merck of Darmstadt, from Kahlbaum of Berlin, and commercially from France, Raikow finds that it is absolutely free from sulphur, and that the red colour is due to an ingredient containing chlorine. If a little cotton oil is burnt in the tubular torch already described (*loc. cit.*), and the flame is brought inside a beaker charged with a few drops of potassium hydroxide solution, which is held horizontally and slowly rotated so that its internal surface is wetted all over, the liquid will finally give the reactions of hydrochloric acid, but not those of sulphuric acid. Similarly, a repetition of Dupont's experiments (extraction of the oil with steam) has yielded distillates quite free from sulphuric acid. The chlorine compound in the original cotton oil is perfectly insoluble in water and alcohol, is not affected by treatment with steam at 100° C., but is slightly volatile in highly superheated steam; whether it can be entirely removed from the oil in this manner remains to be seen. Of the oils which give no colour with phloroglucinol and vanillin (*ut sup.*), olive and walnut contain neither sulphur nor chlorine; of those which do give a red colour, colza and rape contain sulphur but no chlorine.

According to Benedikt. the presence of chlorine in fats is due to carelessness in carrying out a bleaching process, but Kilgore has stated that American cotton oil is bleached without the use of halogen. The chlorine must therefore be a natural constituent, and if it exists in every variety of cotton oil, a characteristic test for the material is thus given. Either the phloroglucinol-vanillin reaction or the test with potassium hydroxide may be employed to seek for the halogen; but if only a little cotton oil be present in the sample under examination, it is necessary to burn several c.c. In such cases the flame is best held under a funnel, which is connected with a set of bulbs containing the alkali, and thence with a pump, a blank experiment being conducted at the same time to insure purity of the potash and absence of hydrochloric acid vapours in the laboratory atmosphere. Combustion of 1 c.c. of an olive oil mixed with 6 per cent. of cotton oil is sufficient to yield an indication of chlorine; if the former contains only 1 per cent. of the adulterant, 5 c.c. must be burnt.

As the whole of the chlorine or bromine in an organic body is converted by

ignition into hydrochloric or hydrobromic acid, the percentage of halogen in any combustible organic substance may be determined by operating in this manner.

F. H. L.

[No reference is made to Charabot and March's recent investigation (ANALYST, 1899, xxiv., 247), in which the existence in cotton oil of Dupont's volatile sulphur compound was corroborated, and in which also the analogous sulphur compound in olive oil discovered by Dupont and Charabot was again mentioned.—ABS.]

THE GOVERNMENT LABORATORY AND MINERAL MATTER IN TEA.

(*From the "Derby and Chesterfield Reporter," April* 28, 1899.)

CHARGE OF SELLING ADULTERATED TEA.

AT the Derby County Police Court on Friday morning, before Mr. S. Leeke (in the chair), Mr. W. Mallalieu, and Mr. E. S. Milnes, William Henry Woodward and Edgar Woodward, grocers, of Draycott, were summoned for selling 4 ounces of caper tea which was adulterated with at least 6 per cent. of ferruginous sand and small stones at Draycott on January 23. Mr. R. S. Clifford appeared to prosecute, and Mr. Bendle Moore conducted the case on behalf of the defendants.—Mr. Clifford said the formal portion of the evidence had been taken on February 24, and Mr. Moore then made an application that the third sample should be sent to Somerset House for analysis. The Somerset House analysis completely bore out the analysis of Mr. White, the County Analyst, viz., that there was 6 per cent. of mineral matter in the tea.—. . . Mr. E. Lewin, Superintendent Analyst at Somerset House, said . . . he did not agree with Dr. Bell's statement in his book on the subject of adulteration. *A point that could not be lost sight of in this case was the fact that the tea had passed the Custom House Inspector.* Mr. Lewin went on to explain the method of dealing with tea at the Custom House, adding that such particular attention was paid to that article by the inspectors and tea-tasters that it really was not worth the time of the Public Analyst to trouble about it at all. *The tea would have been deemed by the Custom House Inspectors to be commercial tea. He believed the inspectors after analysis would have deliberately passed the tea as fit for home consumption.* So long as tea passed the Customs he did not think the Public Analyst had much to do with it.—Cross-examined by Mr. Moore: Mr. Lewin said that the amount of silica and sand in the tea was either natural to the tea itself, or became mixed in the course of preparation. *The analyst at the Custom House had passed tea containing more mineral matter than was present in this case.*—After a legal argument as to the bearing of certain other cases upon this one the Bench retired. They returned after an absence of half an hour, and the Chairman said that the Bench were of opinion that the amount of extraneous matter which had been proved to have existed in the caper tea was, upon the evidence, unavoidably mixed in the process of collection, and therefore there would be no conviction.*

The following correspondence has passed between the Hon. Secretaries and the Principal of the Government Laboratory and the Secretary to the Customs:

[Copy.]

June 13, 1899.

DEAR DR. THORPE,

We are instructed by the Council of the Society of Public Analysts to call your attention to the statement made by Mr. Lewin in the course of his evidence in a case of alleged tea adulteration at Derby, to the effect that the analyst at the Customs House would pass as genuine tea containing even more mineral matter than the sample in question, which, according

* The fact that Mr. White found practically the same amount of total ash in the tea that Mr. Lewin did (13·6 per cent.) is not recorded in the above extract.—EDITOR.

to Mr. Lewin, gave 13·6 per cent. of total ash. As this allowance for extraneous matter is, as far as our knowledge goes, larger and more liberal than any hitherto sanctioned, we shall esteem it a favour if you will inform us what limit for total mineral matter, or for extraneous matter beyond the natural ash of tea, is authorised and adopted by the authorities of Her Majesty's Customs.

We herewith enclose a copy of the certificate of Messrs. Lewin and Davis, and a newspaper report of the proceedings.

We are, yours faithfully,
(Signed) E. J. BEVAN, A. C. CHAPMAN. } Hon. Secretaries.

Dr. T. E. THORPE, F.R.S.

GOVERNMENT LABORATORY, *June* 20, 1899.

DEAR SIRS,

I beg to acknowledge the receipt of your letter of the 13th inst.

As you are doubtless aware, the chemical examination of the tea imported into the United Kingdom is, by direction of Clause 30 of the 36 and 39 Vict., undertaken by persons appointed by the Commissioners of Customs, subject to the approval of the Treasury. I have not been called upon by the Commissioners to fix the limits for total mineral matter, or for extraneous mineral matter beyond the natural ash of tea, which determine when a sample of tea shall not be allowed to pass into consumption in this country.

I must, therefore, refer your Council to the Comissioners of Customs for information as to the limits which are adopted and authorized presumably by the Treasury.

Yours very truly,
(Signed) T. E. THORPE.

E. J. BEVAN, ESQ., A. C. CHAPMAN, ESQ., } Hon. Secretaries.

LONDON, *June* 28, 1899.

SIR,

We are instructed by the Council of the Society of Public Analysts to call your attention to a statement made in a recent case of alleged tea adulteration at Derby by Mr. Lewin of the Government Laboratory, to the effect that the Customs officials were in the habit of passing as genuine tea containing more than 13·6 per cent. of ash.

We shall esteem it a favour if you will kindly inform us if this statement is correct, and what is the limit adopted.

The matter is one of considerable importance to Public Analysts. We herewith enclose a newspaper report of the proceedings.

We are, your obedient servants,
(Signed) E. J. BEVAN, A. C. CHAPMAN, } Hon. Secretaries.

RICHARD T. PROWSE, ESQ., C.B.

CUSTOMS HOUSE, LONDON, *June* 30, 1899.

GENTLEMEN,

I have to acknowledge the receipt of your letter, dated the 28th inst., relative to the inspection of imported tea by officers of this Department, and to say that the subject shall have early attention, and a reply be sent to you in due course.

I am, Gentlemen, your obedient servant,
(Signed) R. T. PROWSE.

THE HON. SECRETARIES,
Society of Public Analysts.

CUSTOMS HOUSE, LONDON, *July* 5, 1899.

GENTLEMEN,

I am directed by the Board of Customs to acquaint you, with further reference to your

letter of the 28th ult., that they are informed by their official analyst for tea that the records for the past two and a half years have been carefully searched, with the result that no instance can be traced in which tea which has proved on analysis to contain as much as 13·6 per cent. of ash has been allowed to pass into consumption.

I am, Gentlemen, your obedient servant,

The Hon. Secretaries, (Signed) R. T. Prowse.
Society of Public Analysts.

July 19, 1899.

Sir,

We beg to thank you for your letter of the 5th inst.

We should feel still further obliged if you would kindly let us know, for the information of the Society, the limits for total mineral matter in teas adopted in the laboratory of Her Majesty's Customs, and if possible the time during which those limits have been in force.

We are, your obedient servants,

(Signed) E. J. Bevan, A. C. Chapman, } Hon. Secretaries.

Richard T. Prowse, Esq., C.B.

Customs House, London, *August* 9, 1899.

Gentlemen,

In reply to your further letter of the 19th ult., inquiring as to the limits applied in the laboratory here for total mineral matter in teas, and the time during which such limits have ruled, I am directed by the Board of Customs to acquaint you that, assuming your inquiry to relate to green teas and "capers," it is their general practice to admit such teas for home use when they are found to contain not more than 2 per cent. of facing matter, but that caper teas vary so much in quality and other characteristics that it has not been found practicable to fix a hard-and-fast line in respect of the ash or mineral matter they may be found to contain, each importation being treated on its merits, and that this has been the practice observed from the time when the duty of inspecting and analyzing imported teas was first imposed on the Department.

I am, Gentlemen, your obedient servant,

The Hon. Secretaries, (Signed) R. T. Prowse.
Society of Public Analysts.

Derby Tea Case.

4, New Court, *October* 24, 1899.

Dear Dr. Thorpe,

We have been instructed by the Council of the Society of Public Analysts to forward to you a copy of the correspondence we have had with the Customs authorities on the question of mineral matter in caper teas.

You will see that as far as Mr. Prowse's information carries us the statements made by Mr. Lewin are not supported.

We are, yours faithfully,

(Signed) Edward Bevan, Alfred C. Chapman, } Hon. Secretaries.

Dr. T. E. Thorpe, F.R.S.

Government Laboratory, *October* 27, 1899.

Gentlemen,

I am directed by Dr. Thorpe to acknowledge the receipt of your letter of the 24th inst., and I am to state that he is much obliged to you for the information you are so good as to send him.

Dr. Thorpe observes that the Board of Customs are of opinion that it has not been found

practicable to fix a hard-and-fast line in respect of the ash or mineral matter that caper tea may be found to contain.

He thinks, however, it would have been more pertinent to the point at issue if you had elicited from the Board of Customs whether the particular caper tea in question was or was not adulterated.

I am, Gentlemen, your obedient servant,

Messrs. BEVAN AND CHAPMAN, (Signed) F. J. REED.
Hon. Secretaries,
Society of Public Analysts.

4, NEW COURT, *November* 8, 1899.

DEAR DR. THORPE,

We are much obliged for your favour of the 27th ult., which was laid before the Council on the 1st inst.

We are instructed by the Council to say that so far as appears from the information given by the Customs authorities, Mr. Lewin's statement to the effect that the Customs were in the habit of passing teas containing 13·6 per cent. of total mineral matter is not warranted.

In view of the importance of the matter to Public Analysts, the Council proposes to publish the correspondence.

We are, yours faithfully, etc.

(Signed) E. J. BEVAN, A. C. CHAPMAN, Hon. Secretaries,

Dr. T. E. THORPE, F.R.S.

Mineral Matter in Tea.

LONDON, *November* 8, 1899.

SIR,

The information which you have been good enough to furnish us with on this subject has been laid before the Council, and we are instructed to inform you that in view of the importance of the matter to Public Analysts, it is proposed to publish the correspondence.

We are, yours obediently,

(Signed) E. J. BEVAN, A. C. CHAPMAN, Hon. Secretaries.

RICHARD T. PROWSE, ESQ., C.B.

CUSTOM HOUSE, LONDON, *November* 18, 1899.

GENTLEMEN,

I am directed by the Board of Customs to acknowledge the receipt of your letter of the 8th inst., and to state in reply that, so far as they are concerned, they do not offer any objection to the proposal to publish the correspondence referred to in that letter.

I am, Gentlemen, your obedient servant,

The HON. SECRETARIES, (Signed) R. T. PROWSE.
Society of Public Analysts,
4, New Court, Lincoln's Inn, W.C.

ERRATA.

Page 292, seventeenth line from bottom, and all through abstract, *for* "cresol" and "homocresol" *read* "creosol" and "homocreosol."

Page 302, eighth line, *for* "tin" *read* "zinc."

The Analyst,

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Society of Public Analysts.

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THE ANALYST.

JANUARY, 1900.

OBITUARY NOTICE.

THE LATE PROFESSOR HODGES, M.D., F.I.C., Etc.

It is with regret that we have to chronicle the death, on Dec. 13, 1899, at Belfast, of Professor Hodges, who had attained the venerable age of eighty-four years, and was a distinguished member of our society. He was born and educated at Downpatrick, and afterwards studied medicine at Dublin and Glasgow, qualifying at both places. During his stay at the latter city Mr. Hodges made the acquaintance of Professor Graham, who strongly advised him to devote his talents exclusively to chemistry. Mr. Hodges, however, preferred to adhere to his original intention, and commenced the practice of medicine at Newcastle, afterwards removing to Downpatrick. He there evinced much ability as a lecturer, and became so deeply interested in agriculture that this induced him to proceed to Giessen to study chemistry under Liebig, where his ability and diligence so interested the Baron that the two became intimate friends. At Giessen he took the degree of Doctor of Medicine. Dr. Hodges then returned to Downpatrick, and there delivered a lecture on agriculture, which attracted so much attention that one of its results was the formation of the Chemico-Agricultural Society of Ulster, in 1845, to which Dr. Hodges was appointed chemist. He removed to Belfast in 1844, and shortly afterwards was appointed Professor of Chemistry in the old Belfast College. On the foundation of a Chair of Agriculture in Queen's College, Belfast, Dr. Hodges was chosen as its first occupant, and also as Lecturer on Medical Jurisprudence there. Both of these positions he held to the day of his death. He was also Lecturer on Agriculture at the Glasnevin Agricultural School. Dr. Hodges wrote many treatises and papers, which had a wide circulation. He was one of the founders of the Royal College of Chemistry, London. He held many important offices. For a long time he was Government Analyst, also Public Analyst for Belfast, and for five Irish counties. He was a corresponding member of the Dublin Natural History Society, member of the Royal Academies of Agriculture of Sweden, and of Turin, and of the Imperial College of Gorygoretzt, Russia; also a Justice of the Peace for County Antrim.

PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS.

THE monthly meeting of the Society was held on Wednesday evening, December 6, in the Chemical Society's Rooms, Burlington House, the President (Mr. W. W. Fisher, M.A.) occupying the chair.

The minutes of the previous meeting were read and confirmed.

Certificates of proposal for election to membership in favour of Messrs. J. A. Foster, J. B. P. Harrison and D. Lloyd Howard were read for the second time; and a certificate in favour of Mr. Bertram William John Warren, A.I.C., assistant to Mr. Otto Hehner, was read for the first time.

Messrs. E. C. P. Barber, Frederick Davis, William Francis, Alfred Lucas, Edward Russell, B.Sc. (Lond.), and F. L. Slocum, Ph.D., were elected members of the Society.

On the proposition of Mr. Ekins, seconded by Mr. Richmond, Mr. Benedict Kitto and Mr. Cecil H. Cribb, B.Sc., were appointed to act as auditors of the Society's accounts for the year 1899.

The following papers were read: "On some Analyses of Modern Dry Champagnes," by Otto Rosenheim, Ph.D., and P. Schidrowitz, Ph.D.; "Note on Asafœtida," by J. M. Martin, B.A., M.B., and C. G. Moor, M.A.; "On the Determination of the Iodine Value," by Dr. J. J. A. Wijs; "Treacle or Golden Syrup," by E. W. T. Jones; and "On a Method for distinguishing between Hops and Quassia," by Alfred C. Chapman.

NOMINATION OF OFFICERS AND NEW MEMBERS OF THE COUNCIL FOR 1900.

The following nominations were made by the Council at their meeting on December 6, 1899:

President.—W. W. Fisher, M.A. *Vice-Presidents.*—Charles E. Cassal, F.I.C.; E. W. T. Jones, F.I.C.; S. Rideal, D.Sc. *Hon. Treasurer.*—E. W. Voelcker, A.R.S.M. *Hon. Secretaries.*—E. J. Bevan, F.I.C., A. C. Chapman, F.I.C. *Other Members of the Council.*—L. Archbutt, F.I.C.; A. Ashby, M.B., F.R.C.S., F.I.C., G. Embrey, J. H. B. Jenkins, P. A. E. Richards, F.I.C., H. D. Richmond, F.I.C.

NOTE ON ASAFŒTIDA.

BY J. M. MARTIN, B.A., M.B., AND C. G. MOOR, M.A.

(*Read at the Meeting, December* 6, 1899.)

A SAMPLE of gum-resin of asafœtida having been found to yield a very much higher ash than the standard given by the British Pharmacopœia, a number of samples were examined to determine the general condition of the commercial article.

Twelve samples were examined as to:

1. Matter soluble in 90 per cent. alcohol.
2. Ash of the gum.
3. Portion of ash soluble in HCl.

In all the samples, on grinding, much mineral matter was found, in one case a stone weighing 3·4 grammes.

The percentage of the gum soluble in alcohol (90 per cent.) ranged from 14·8 to 39·8, instead of the 65 per cent. of the British Pharmacopœia.

The ash of the gum ranged from 63·1 to 26·4 per cent., the British Pharmacopœia figure being 10 per cent.

The solubility of this ash in 50 per cent. HCl varied from 15·7 to 57·9 per cent., but in one case reached 96·4 per cent., nearly the whole going into solution with much effervescence.

The British Pharmacopœia directions for making the tincture of asafœtida are to extract 200 grammes of the gum-resin with 1 litre of 70 per cent. spirit. Hence, supposing 65 per cent. of the gum were soluble, the tincture should contain 13·5 grammes per 100 c.c. of total solids. Seven tinctures were examined for total solids, and gave results ranging from 4·5 to 8·5 grammes per 100 c.c.

The results of the analyses made are as follows:

Sample.	Extract.	Ash of Gum.	Ash insoluble in HCl.
I.	31·9	38·6	82·5
II.	30·2	45·8	3·6
III.	14·9	62·1	77·8
IV.	20·2	56·3	84·3
V.	18·4	59·3	74·2
VI.	35·4	40·1	64·9
VII.	19·9	58·4	74·6
VIII.	23·0	52·9	78·2
IX.	14·8	63·1	83·9
X.	39·8	43·4	42·1
XI.	37·7	26·4	67·2
XII.	28·9	47·2	78·4

Tinctures.

I.	8·2 grammes per 100 c.c.	V.	5·3 grammes per 100 c.c.
II.	6·2 ,, ,,	VI.	4·3 ,, ,,
III.	4·5 ,, ,,	VII.	8 5 ,, ,,
IV.	7·6 ,, ,,		

It is, then, evident that commercial samples of the gum-resin of asafœtida are very far below the standard given by the British Pharmacopœia, and that they are probably intentionally weighted with worthless material. Consequently, preparations made from the gum-resin according to the British Pharmacopœia directions fall far below the presumed strength, and the medical profession are prescribing medicines of uncertain and variable composition.

A few other observers have noted this condition of commercial gum-resin of asafœtida, yet but little attention seems to have been given to their results.

Moerner (*Journ. Pharm. Chim.*, March 1, 1888, p. 24) examined a sample, and found 86 per cent. of mineral matter, and only 14 per cent. asafœtida. A large proportion, on fracture, proved to be alabaster coated with a thin layer of asafœtida resin.

Buckner (*A. J. P.*, 1890) found the ash in a number of samples to range from 19 to 56 per cent.

Lloyd (*P. J.*, i., 243, 1896) said "ash is often higher than the 10 per cent. required by the British Pharmacopœia." The *National Dispensatory* states that the ash of commercial samples is sometimes as high as 40 per cent.

A thirteenth sample was examined which had been specially prepared by a firm of chemists with a view to determining how far it was practicable attempting to produce a sample approaching British Pharmacopœia strength from this raw material. They succeeded in increasing the matter soluble in 90 per cent. alcohol to 57·5 per cent. (British Pharmacopœia, 65 per cent.). The ash of this was only 5·8 per cent.

Although asafœtida in its various preparations is now prescribed somewhat less than formerly, yet we certainly ought to have reliable material. The standard of the British Pharmacopœia is too high, unless means can be taken to prevent adulterated resin reaching the market. In this connection it may be remembered that a few years ago many samples of ginger were discovered with an abnormally low soluble ash, and that when attention had been drawn to this, and a few prosecutions followed, such ginger almost entirely disappeared from the market.

Discussion.

The President having invited discussion,

Mr. Chattaway said that the British Pharmacopœia, in addition to laying down certain regulations as to the proportion of ash, solubility in alcohol, etc., described the physical characters of the drug; and it would be interesting to know if the samples referred to were in accordance with the Pharmacopœia in this respect.

Mr. E. M. Holmes said that about a year previously, in his capacity as one of the referees in connection with the compilation of the present Pharmacopœia, he had had analyses made of several samples of asafœtida. He was aware that the commercial form of the drug was largely mixed with mineral matter, but, at the same time, the gum in the form of "tears" was almost pure, and he had experienced no difficulty in obtaining it. When the gum was being collected, a certain number of the tears were obtained pure, except for a small quantity of sand attached to their lower surface, where they fell on the ground; but there was a large quantity of more or less liquid asafœtida, which could not be collected in the ordinary way, and this was mixed by the collectors of the drug with a certain quantity of whatever soil might be obtainable in the neighbourhood in order to solidify it. Sulphate of lime, in the form of alabaster, was probably used as an adulterant, not only for asafœtida, but for benzoin, in which it could be used to represent white tears, a certain proportion of which was normally present in the lump form of the drug. In the samples which had been analysed for him the ash in no case exceeded 8 per cent., but in the crude material in lumps it often ranged from 20 to 60 per cent. He thought that, while for medicinal purposes a drug as pure as possible was desirable, a commercial article was admissible which could be used for drain-testing, or for denaturing tea in the manufacture of caffeine; and a normal standard might be laid down for the crude drug, quite apart from the medicinal point of view. The supply of asafœtida sufficiently pure for medicinal purposes was limited; but there was a fairly abundant supply of the ordinary form, in which a moderate proportion of mineral matter—say, from 20 to 30 per cent.—was for commercial purposes not of much consequence,

seeing that for such purposes the article would be bought on the basis of its percentage of active matter.

Mr. ALLEN said he felt some difficulty in agreeing with Mr. Holmes's suggestion to establish two qualities of the drug. The addition of calcium sulphate in the case of the lump asafœtida was an intentional adulteration, which it would, he thought, be dangerous to countenance. Even though the adulterated material were intended only for commercial purposes, he did not see why the purchaser should be supplied with calcium sulphate when he might imagine that he was getting pure asafœtida.

Mr. CRIBB said that he had found the ash in asafœtida to vary from 15 to 35 per cent., and the proportion soluble in alcohol was correspondingly low. The samples were purchased indiscriminately in London. As far as he could ascertain, the drug in the solid form was hardly ever asked for except in remote country districts, and it was now rarely employed in medicine in any form.

Mr. EKINS said that the drug was very seldom used in the solid form except for veterinary purposes, and the prescriptions in which the tincture appeared were very few.

Mr. MOOR said that the samples he had examined were all in the form of a hard concrete mass, which was the only form of the drug to which his experience extended. Regarding the tincture, which was the chief form in which the drug was used medicinally, it was perfectly easy to make the tincture up to the proper standard by taking a suitable quantity of asafœtida.

Dr. RIDEAL observed that in the Pharmacopœia it was directed that, in making the tincture and fetid spirit of ammonia, a certain quantity of the drug should be taken, and it seemed open to question whether the use of a less pure drug in larger quantity would be in strict conformity with the directions.

Mr. MOOR said that to sell a tincture of as nearly as possible the proper strength —even though it might be made from a larger quantity of the drug than the Pharmacopœia directed—would, he thought, be better than deliberately to sell a weak tincture, and accordingly he would suggest that the tincture itself should be standardized. It would be unreasonable, he thought, too strongly to insist on conformity with the British Pharmacopœia tests, and he would suggest that for the present asafœtida containing not more than 20 per cent. of ash, and not less than from 45 to 50 per cent. of matters soluble in alcohol, should be accepted.

Mr. CHATTAWAY said that the Pharmacopœia laid down that the drug should be in the form of tears—indeed, the actual size of the tears is specified; and he thought that the specimens referred to by the author, although they might be admissible commercially, could not be regarded as representing the British Pharmacopœia article, which frequently contained as little as 2 or 3 per cent. of ash.

Mr. HOLMES said that, while the British Pharmacopœia limit of ash was 10 per cent., a much smaller percentage was quite common in the drop or tear asafœtida. He thought that lump asafœtida would be admissible only provided that it contained less than 10 per cent. of ash. The question of purity was merely a question of the price given for the drug, which varied from about 45s. or 60s. for the lump, to 120s. per cwt. for the finest tears. Seeing that the Pharmacopœia intimated that only the pure drug should be used in medicine, he did not think it would be justifiable to use

the drug in an impure state in medicine, and in its purification some volatile active ingredients might be lost, and the British Pharmacopœia standard of strength would not be maintained.

ON SOME ANALYSES OF MODERN "DRY" CHAMPAGNE.

By Otto Rosenheim, Ph.D., and Philip Schidrowitz, Ph.D.

(*Read at the Meeting, December* 6, 1899.)

The authors have recently had an opportunity of examining a number of champagnes representative of the modern "dry" French product, and as there are practically no analyses of similar wines on record, we think it may be of interest to communicate the results obtained to the Society.

The figures obtained by the analysis of wines are to a great extent—as is the case with many other articles of food—dependent on the methods employed, and therefore we thought it advisable to use the standard methods officially adopted in Germany,* in order to be able to compare our results with the data on this subject already in existence. The official German schedule was drawn up as a guide in the analysis of still wines, and therefore no mention is made of the estimation of carbonic acid. We determined the same gravimetrically by absorbing the gas in soda lime after drying with sulphuric acid in the ordinary manner, the last trace being expelled by heating in a water-bath, and then passing a current of air through the liquid. In order to regulate the flow of gas and to prevent frothing over, we made use of one of the well-known hollow champagne corkscrews with an accurately-fitting tap.

On the opposite page is a table of the analytical results obtained.

With regard to the composition and manufacture of champagne, Grünhut (*Z. f. analyt. Chem.*, 1898, 231) recently put forward the suggestion—based solely on analytical figures—that this article in general bears the character of a "gallisised" wine—that is, a product which has been unduly diluted by the addition of a weak solution of sugar prior to fermentation, and that this is principally evidenced by the low percentage of ash. The figures we have obtained appear at first sight to corroborate Grünhut's conclusions, to which he came, however, by an erroneous method of reasoning. He compared his figures with averages obtained from a large number of analyses of German white wines, but there is no justification for this, as the methods of manufacture are so completely different that no strict comparison can be made between the two classes of wine. Practically all better-class champagne is made from blue grapes, and in order to meet the exigencies of the market, which demands a very light-coloured article, the grapes must not be subjected to any preliminary crushing or kneading, and special care is taken to ensure a rapid separation of the juice from the skins, pips, etc., in the press. This procedure, adopted for the purpose of obtaining a light-coloured must, necessarily tends to prevent the extraction of mineral matter from the skins, stalks and pips, and it is therefore to be expected that the finished article will contain a relatively small percentage of ash. In the manufacture of ordinary white wines a preliminary crushing takes place in the vineyard,

* German Wine Law, June 25, 1896.

ANALYTICAL RESULTS.

No.	Designation.	Vintage.	Specific Gravity at 15° C.	Rotation in 200 mm. Tube.	Alcohol per Cent. by Weight.	1,000 c.c. contain Grammes.									Extract – (Sugar – 1).	Reduced Extract : Ash :: 100 : x.
						Free Acid (calculated to Tartaric Acid).	Fixed Acid (calculated to Tartaric Acid).	Volatile Acid (calculated to Acetic Acid).	Extract.	Ash.	Total Tartaric Acid.	Sugar (as Invert Sugar).	Glycerin.	Carbonic Acid.		
1.	Pommery and Greno. Champagne nature	1892.	0·9904	–0·23°*	11·11	5·22	4·57	0·52	20·95	1·17	2·20	3·36	7·55	8·27	18·59	6·29
2.	Clos des Cordeliers. Reserve, brut†		0·9958	–0·38°	9·87	4·03	3·43	0·48	28·56	0·97	3·99	10·61	6·28	8·85	18·95	5·12
3.	Veuve Cliquot Ponsardin, brut ...		0·9909	–0·10°	9·97	3·23	2·71	0·42	19·78	2·52	2·76	1·32	7·64	7·79	19·46	12·91
4.	Veuve Monnier et ses Fils. Grand vin, extra sec		0·9989	–0·75°	9·96	6·89	6·27	0·49	37·67	1·27	2·24	19·50	9·25	6·35	19·17	6·61
5.	Heidsieck Dry Monopole, special cuvée		0·9939	–0·38°	10·72	5·99	5·35	0·51	27·07	1·16	2·10	9·20	9·10	9·55	18·87	6·14
6.	Binet Fils et Cie. Dry élite ...		0·9929	–1·00°	11·87	3·47	2·81	0·53	27·20	1·27	2·28	9·88	6·77	7·86	18·32	6·93
7.	Pommery et Greno. Extra sec ...	1893.	0·9911	–0·48°	10·74	5·01	4·32	0·56	22·95	1·10	2·18	7·84	6·50	8·12	18·11	6·82
8.	Perrier et Jouet. Extra dry ...		0·9927	–0·15°	9·96	5·43	4·67	0·59	23·18	1·13	2·49	7·23	8·18	7·75	16·95	6·66
9.	Jules Mumm et Cie. Extra dry		0·9942	–0·75°	10·96	6·03	5·28	0·60	29·14	1·22	2·45	11·87	7.76	8·14	18·27	6·87
10.	Renaudin, Bollinger et Cie. Extra quality, very dry		0·9897	–0·10°	11·40	3·84	2·78	0·85	19·99	1·56	2·56	3·77	5·92	10·21	17·22	9·05
11.	Laurent Perier et Cie. Grand vin sans sucre		0·9930	–0·61°	10·79	6·26	5·54	0·58	25·23	1·15	2·37	8·44	6·68	9·43	17·79	6·46
12.	Veuve Cliquot Ponsardin, dry ...		0·9940	–0·66°	11·45	4·80	4·18	0·50	30·33	1·05	2·04	13·86	9·05	—	17·47	6·01
13.	Pfungst Frères et Cie. Ay-champagne, Carte d'Or		0·9934	–0·66°	11·04	6·35	5·77	0·46	26·39	1·25	2·94	8·14	6·62	—	19·25	6·49

* The readings were made in a Laurent half-shadow apparatus at 15° C.

† This wine is not actually a "champagne," but is made according to the methods of the Champagne from blue grapes in St. Émilion, Gironde.

and the separation of the juice from the marc is not hastened as in the preparation of champagne. In connection with the above remarks, we may add that similar views were expressed by Kulisch (*Z. f. angew. Chem.*, 1898, 573) in criticising Grünhut's paper.

With regard to our own results, it will be observed that both the extract and the ash generally are rather low (compared with Bordeaux or Rhine wines), with the exception of No. 4 (3·76 per cent.), which is due to the high percentages of sugar and glycerin. No. 3 shows a high percentage of ash. In regard to the sugar percentages, it will be seen that only Nos. 4, 9 and 12 are appreciably above 1 per cent. The relation of "reduced extract" to ash is considerably below 10 per cent. (that being the normal average deduced from a large number of analyses of still white wines), except in the case of Nos. 3 and 10. We have already given an explanation for this apparent anomaly, which constitutes an excellent illustration of the fact that it is in many cases extremely dangerous and ill-advised to judge the quality and value of an article of food solely in the light of the figures obtained by analysis.

No. 10 shows some features of interest; it is the only case in which the specific gravity falls below 0·99, but this is probably due to the high percentage of alcohol in relation to the low extract. The total acidity is small, but the volatile acid relatively large. This sample also showed the highest percentage (1·02) of carbonic acid.

Another noteworthy feature is the high lævo-rotation of No. 6, which indicates a preponderance of lævulose. The absence of cane-sugar was demonstrated by inversion, which did not alter the rotation. According to Neubauer (*Ber.*, x., 827) the proportions of dextrose and lævulose in a mixture can be calculated according to the following equations:

$$1.\ X = 0{\cdot}637S + 0{\cdot}342\alpha.$$

$$2.\ Y = S - X.$$

X = dextrose; Y = lævulose; S = total invert sugar found; α = rotation observed in 200 mm. tube.

Although the results obtainable from these equations are not absolutely correct (for reasons into which we cannot enter here), they are sufficiently exact to permit of the approximate determination of the proportion of dextrose to lævulose respectively. On applying the above formulæ to the case in point, it was found that the quantity of lævulose was 7·01, and of dextrose 2·87. In most cases the proportion is as 1 : 1, and this indicates that in No. 6 the fermentation of the sugar in the added liqueur ceased after the disappearance of the greater part of the dextrose.

Discussion.

The President observed that, speaking broadly, it seemed that the proportion of extract in those cases in which it was high rose with the proportion of sugar, which probably depended on the composition of the liqueur that was added to the grape-juice after fermentation. He presumed that there might still remain a certain quantity of unaltered cane sugar, though doubtless most of the sugar would be inverted during secondary fermentation.

Dr. Schidrowitz said that in those cases in which the determination had been

made the sugar present was found to be completely inverted. It would become inverted very quickly, chiefly owing to the acidity of the liquid in question.

Mr. RICHMOND remarked that in a liquid which had undergone fermentation by means of yeast it was unlikely that there would be any unaltered cane sugar present, complete inversion being brought about by an enzyme secreted by the yeast.

The PRESIDENT observed that before the liqueur was added primary fermentation would be over.

Mr. RICHMOND said that the enzyme would still be present in the yeast cells remaining in the wine, and the sugar in course of time must, he thought, surely become inverted.

Mr. CRIBB inquired where a description of the German official methods of analysis was to be found.

Dr. SCHIDROWITZ said that the official methods were embodied in the German Wine Law, dated June 25, 1896, and were to be found in the appendices to most German works on the subject (*e.g.*, Windisch, *Die chemische Untersuchung u. Beurtheilung des Weines*, p. 45 and *e*).

ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

FOODS AND DRUGS ANALYSIS.

The Occurrence of Chromates as Preservatives in Milk. A. Leys. (*Journ. Pharm. Chim.*, 1899, x., 337-340.)—During the present year the author has frequently met with chromates associated with formaldehyde, most frequently in the proportion of 1 part in 100,000 of milk, though considerably larger quantities are of not unfrequent occurrence.

In order to detect traces of chromates the following tests are recommended: From 100 to 150 c.c. of the milk are evaporated to dryness, the residue ignited, and the ash extracted with a few c.c. of water, note being taken of the colour of the solution.

A reagent is prepared by adding a few drops of indigo carmine solution to concentrated hydrochloric acid until the liquid has the colour of Fehling's solution. On boiling 5 c.c. of this reagent with a few drops of the filtered aqueous extract of the ash, the colour is instantly discharged in the presence of the slightest trace of chromate.

A second test consists in adding some pure aniline and commercial toluidine to an excess of acetic acid free from furfural, filtering through purified animal charcoal, and diluting the filtrate to the tint of the extract of milk ash. On boiling equal parts of this reagent and the extract for two or three minutes, a cherry-red colour is produced after a short time in the presence of a chromate.

The two reactions described above are said to be much more sensitive than Schiff's guaiacum reaction for chromic acid, and point to the presence of an oxidizing agent. In order to identify this as a chromate Barreswil's hydrogen peroxide reaction may be employed. The aqueous extract of the ash is acidified with a few drops of dilute sulphuric acid and two or three drops of hydrogen peroxide added. The immediate formation of the fugitive blue tint of perchromic acid indicates the presence of a chromate. C. A. M.

On Fruit Juices and their Examination, with particular reference to Raspberry Juice. I. Detection of Foreign Colouring Matter. E. Spaeth. (*Zeit. für Untersuch. der Nahr. und Genussmittel*, 1899, ii., 633-635.)—The author deals principally with the detection of artificial colouring matter in raspberry juice. One of the principal means of sophistication is by admixture with cherry juice, and though the lead acetate method is said to provide a means for the detection of even small quantities, the author cannot confirm this, nor is he able to indicate any method at present by which admixture with cherry juice can be detected with certainty. Other vegetable colouring matters used for the purpose are those of the whortleberry, red beet, red poppy, mallow, phytolacca and orseille. Cochineal is also used. Of these, orseille, phytolacca, beet and cochineal can be identified, whilst in a general way the addition of the juices of such other fruits, as the whortleberry, the blackberry, and the elderberry, can be detected, though in most cases it is impossible to say which of these has been added, their behaviour with reagents being so similar. Of the tar colours a large and ever-increasing number is made use of, such as rosaniline, fuchsin, eosin, etc.

The table opposite gives the principal reactions of the various colouring matters which the author has studied:

Tar colours dissolved by amyl alcohol can be distinguished from vegetable colours by the colour being removable by means of wool freed from fat. S. fuchsin, however, not being soluble in amyl alcohol, it is necessary also to submit the diluted juice itself to the wool test.

In the sophistication of lemon-juice, tar colours are always used, and the amyl alcohol test with the removal of the colour by wool is almost exclusively available. H. H. B. S.

The Detection of Saponin in Lemonade. Frehse. (*Journ. Pharm. Chim.*, 1899, x., 13-16.)—In a communication on the composition of the lemonade manufactured in Lyons in 1898, the author states that from time to time he has found saponin, but that its use is not general, owing to its being readily precipitated and causing a turbidity in the liquid.

It can be recognised by the persistent foam produced on shaking the bottle after the expulsion of the carbon dioxide, but its chemical identification is not easy in the presence of sugar and tartaric acid. The tests based on its conversion into

Amyl Alcohol.			Other Solvents.	Lead Acetate.	Lead Hydroxide.	Mercuric Chloride.	Ferrous Sulphate.	Aluminium Acetate and Sodium Carbonate.	Ammonium Alum.	Ammonia.	Alkali (Na_2CO_3 KOH).	Acids.	Sodium Phosphate.	Chalk.	Magnesia, or Magnesium Carbonate.
Direct.	From Acid Solution.	From Alkaline Solution.													
—	Pale yellowish.	—	—	Bluish-gray precipitate and filtrate.	Reddish filtrate.	Decolorized.	Reddish.	Precipitate and filtrate gray.	Light red.	Brown.	Brown.	Red.	Decolorized.	Gray.	Grayish-green.
—	—	—	—	Bluish-gray.	Reddish-yellow.	Ditto.	Reddish.	Ditto.	Ditto.	Yellowish-green.	Yellowish-green.	Red.	Ditto.	Yellowish-orange.	Gray.
—	—	—	—	Similar to raspberry, filtrate bluish.	Reddish.	Ditto.	Reddish.	Ditto.	Ditto.	Ditto.	Brownish-green.	Red.	Ditto.	Grayish-red.	Bluish-gray.
Reddish.	Yellowish-red.	—	—	Bluish-green.	Greenish-gray.	Ditto.	Reddish.	Grayish-blue.	Violet.	Greenish-yellow.	Green.	Red.	Ditto.	Grayish-green.	Grayish-green.
Reddish-violet.	Yellowish-red.	—	—	Dark bluish-green precipitate, greenish filtrate.	Ditto.	Ditto.	Reddish-violet.	Ditto.	Violet-red.	Yellowish-green.	Dark green.	Red.	Ditto.	Slate-gray, filtrate greenish.	Green.
Reddish.	Yellowish-red.	—	—	Bluish-green; also filtrate bluish-green.	Ditto.	Ditto.	Bluish-violet.	Ditto.	Ditto.	Green.	Green.	Red.	Ditto.	Grayish-red.	Greenish-blue.
Red.	Red.	—	—	Brownish-gray.	Grayish-green.	Not quite decolorized.	Scarcely decolorized at all.	Gray.	Intense red.	Brownish-green.	Na_2CO_3= yellow-brown; KOH= yellow-green.	Red.	Ditto.	Reddish-gray.	Gray.
Violet-red.	Fine red.	With lead acetate, violet.	—	Fine green.	Fine green.	Decolorized.	Deep bluish-violet.	Bluish-green.	Very fine bluish-violet.	Green.	Green.	Red.	Ditto.	Gray, filtrate green.	$MgCO_3$= green; MgO= green.
—	Pale bluish-red.	—	—	Strongly coloured with separation of a reddish-brown precipitate.	Precipitate of a flesh-coloured red, filtrate yellowish red.	Decolorized, but more slowly.	Nothing characteristic.	Very fine reddish-violet.	Nothing characteristic.	Yellow.	Na_2CO_3= violet; KOH= yellow.	Ruby-red.	Not decolorized.	Filtrate fine red.	$MgCO_3$= violet; MgO= yellowish.
—	—	—	—	Orange-yellow; reddish-yellow precipitate.	Flesh-coloured precipitate.	Decolorized.	Ditto.	Very fine violet-red.	Ditto.	Yellowish-brown.	Na_2CO_3= violet-red; KOH= yellow to yellow-green.	Red.	Ditto.	Ditto.	$MgCO_3$=reddish violet; MgO= yellowish.
Fine red.	Light red.	Lead acetate, reddish-violet.	Direct and acid, fine golden yellow in acetic ether.	Bluish-violet.	Red.	Not decolorized.	Ditto.	Very fine bluish-violet.	Ditto.	Brownish-red.	Na_2CO_3 ruby-red.	Light red.	Ditto.	Reddish-violet.	$MgCO_3$= violet-red-pink.
Yellow.	Yellow.	—	Direct and acid, golden-yellow in acetic ether.	Ditto.	Violet.	Yellow.	Violet.	Reddish-violet-pink.	Ditto.	Ruby-red.	Ruby-red.	Yellow.	Ditto.	Violet-red.	Ditto.
Yellow.	Yellow.	—	The same.	Ditto.	Violet.	Yellow.	Violet.	Ditto.	Ditto.	Ditto.	Ditto.	Yellow.	Ditto.	Ditto.	$MgCO_3$= pink.
—	Very fine yellowish-red.	—	In ether, golden-yellow.	Ruby-red.	Pink.	Decolorized to some extent.	Nothing characteristic.	Very fine reddish-violet.	Ditto.	Violet.	Ditto.	Red.	Ditto.	Pink.	Ditto.
Very fine red.	Very fine red.	Very fine red (also in chloroform).	Direct and acid, in ether, acetic ether, and chloroform, red.	Blue, fine red in chloroform.	Bluish.	Not decolorized.	Bluish-gray.	Greyish-blue.	Reddish violet.	Very fine blue.	Very fine blue.	Red.	Violet.	Very fine blue.	$MgCO_3$= blue.

sapogenin by warming it with hydrochloric acid is uncertain in the case of lemonade. It was not found possible to precipitate it with lead subacetate and to liberate it from the precipitate with hydrogen sulphide, since in the presence of saponin the lead sulphide passed through the filter. Extraction of the lemonade with various solvents is useless, owing to the persistent emulsion produced in every instance, while the influence of the saponin prevents the use of fermentation to remove the sugar.

The only method by which the author could obtain relatively satisfactory results consisted in evaporating the lemonade to a paste and taking up the residue with acetic ether. On filtering this liquid, again evaporating and repeating the treatment with acetic ether, if required, a residue was obtained which gave a reddish-violet coloration with concentrated sulphuric acid, and which when boiled with dilute hydrochloric acid yielded a precipitate of sapogenin, with, as a rule, an odour resembling that of cedar wood. C. A. M.

The Addition of Liquorice Extract to Lemonade. Frehse. (*Journ. Pharm. Chim.*, 1899, x., 347, 348.)—According to the author, extract of liquorice or glycyrrhizin itself have recently been used to produce a persistent froth in lemonade. The reactions given by saponin (see preceding Abstract) are also given by glycyrrhizin, but the latter may be distinguished by its being precipitated in the cold by hydrochloric acid, and by the chlorides of sodium, calcium, and iron.

In the presence of sugar and of glycerin, which is a frequent constituent of foam headings, the reactions are frequently masked. In such cases subacetate of lead is added, and the precipitate filtered off, washed, and decomposed with hydrogen sulphide. In the presence of the tartaric acid of the lemonade the glycyrrhizin is left on the filter with the lead sulphide. The precipitate is washed with water slightly acidified with hydrochloric acid, then rapidly with water, and is taken up with alcohol, and finally with alcohol containing a little ammonia. On evaporating these solutions the glycyrrhizin is left as a yellow residue, readily identified by its odour and its reactions with hydrochloric and sulphuric acids. C. A. M.

ORGANIC ANALYSIS.

The Solubility of Volatile Oils and their Constituents in an Aqueous Solution of Sodium Salicylate. M. Duyk. (*Bull. de l'Acad. Royale de Belg.*, 1899, 503; through *Journ. Pharm. Chim.*, 1899, x., 353-359.)—The solvent power of an aqueous solution of sodium salicylate reaches its maximum when the liquid has a specific gravity of 1·240, and contains 1 part of the salt in 1 of water.

This reagent readily dissolves a large number of alcohols, aldehydes, ketones, and free terpene phenols. On dissolving 1 c.c. of the substance under examination in 4 c.c. of the salicylate solution, and adding water drop by drop, with continual agitation to the solution, the amount required to produce a permanent turbidity

varies considerably with different compounds. Thus, under these conditions, eugenol requires 3·5 c.c.; geraniol and benzaldehyde, 2·5 c.c.; carvol, 2·0 c.c.; citral, 1·7 c.c.; cineol and cinnamic aldehyde, 1·5 c.c.; and citronellone, 0·5 c.c.

Camphor, thymol, and menthol only dissolve in relatively large quantities of concentrated salicylate solution, although the same substances when present in their original oils are much more soluble.

The ethereal compounds of the soluble substances referred to above are not soluble, nor are terpene hydrocarbons, with certain exceptions, such as cymene, limonene, and menthene, which are slightly dissolved owing to the formation of traces of compounds between the terpene and the sodium salicylate.

The author gives the following classification of the principal alcohols, aldehydes, ketones, phenols, etc., of essential oils as regards their solubility in salicylate solution:

SUBSTANCES VERY SOLUBLE:

Alcohols.—Geranylic (rhodinol, geraniol); linalylic (dextro- and lævo-rotatory linalols); citronellic (citronellol); bornylic (borneols); menthylic (menthol).

Aldehydes.—Benzoic; salicylic; anisic; cinnamic; geranic (citral); cuminic; methylpyrocatechuic (vanillin); methylene-protocatechuic (heliotropin).

Ketones. — Carvone (carvol); methylheptenone; pulegone; methylnonylketone; menthone; thujone.

Camphor and cineol.

Phenols.—Eugenol; thymol; carvacrol.

(Menthol and ordinary camphor are soluble with difficulty.)

SUBSTANCES INSOLUBLE OR NEARLY SO:

Sesquiterpene Alcohol.—Santalol.

Phenol Derivatives.—Anethol; safrol; apiol.

Esters.—Bornylic; geranylic; linalylic; menthylic.

In applying this reagent to the examination of essential oils, the sample is shaken with a convenient quantity of the solvent and allowed to stand.

When the oil is composed exclusively of hydrocarbons, the mixture speedily separates into two layers, of which the upper consists of the unaltered hydrocarbon. Pure essence of terebenthene behaves in this way.

If the oil consist of a free oxygen compound, such as oil of bitter almonds (benzaldehyde, with traces of hydrocyanic acid), a clear and limpid solution is obtained.

When an essential oil of complex composition is treated with the reagent the oxygen compounds dissolve, while the esters and hydrocarbons separate out. Thus, in the case of oil of peppermint, the menthol and methone are dissolved, whilst the esters of menthol and menthene remain undissolved.

When the hydrocarbons or other insoluble constituents are only present in small proportion, they may be dissolved through the influence of the soluble oxygen compounds; but on adding an additional quantity of sodium salicylate, the solution becomes turbid, and the hydrocarbon eventually rises to the surface.

To extract the soluble portion of an essential oil by this means, as, for example,

carvol from oil of caraway, 100 grammes of the oil are shaken with 300 c.c. of the reagent for about fifteen minutes, and the mixture allowed to stand until the layers have completely separated, which requires about an hour. The lower layer is then withdrawn, filtered, and mixed with twice its volume of water in a separating funnel. The oily layer which separates is collected, and the aqueous layer concentrated to its original density on the water-bath, and again used for extracting the insoluble portion of the original oil, which will still contain a little carvol.

The carvol can also be recovered from its solution in the sodium salicylate by extraction with ether instead of precipitation with water.

In the case of oils, such as those of peppermint, bergamot, and geranium, which contain alcoholic compounds partly in the free state and partly in the form of esters, one portion of the oil is treated with the salicylate solution as described above to obtain the amount of free alcohols; a second portion is saponified with alcoholic soda, the product treated with water and the liberated oil washed, dried, and treated with the sodium salicylate solution as before to extract the total alcohols present.

In continuation of the subject (*Bull. de l'Acad. royale de Belg.*, 1899, 503; through *Journ. Pharm. Chim.*, 1899, x., 406-411) the author gives, as a first instalment, the following particulars of those essential oils found in the Pharmacopœias:

Oil of Bitter Almonds, which consists of benzaldehyde, with traces of hydrocyanic acid, is completely soluble in 4 volumes of the salicylate solution diluted with 2 volumes of water.

Oil of Absinthe.—The thujone (tanacetone) is readily dissolved; the insoluble residue should not exceed 60 per cent.

Oil of Anise (green).—This is nearly insoluble with the exception of traces of anisic aldehyde.

Oil of Bergamot.—The author found 12 per cent. of soluble constituents by direct treatment, and 35 per cent. after saponification.

Oil of Cajeput.—This is composed of cineol and hydrocarbons. A sample of undoubted purity yielded 53 per cent. of soluble products consisting of nearly pure cineol, whilst cumene was identified in the insoluble residue.

Oil of Cinnamon (Ceylon).—This is completely soluble in the concentrated solution, but is only partially soluble in a mixture of 4 volumes of the reagent with $1\frac{1}{2}$ volumes of water. The proportion of insoluble substances in an oil of good quality should not exceed 10 per cent.

Oil of Cinnamon (China).—On diluting the concentrated solution by one-fourth, the cinnamyl acetate and hydrocarbons are liberated. Their proportion should not exceed 25 per cent.

Oil of Caraway.—On treating this oil with a mixture of 4 volumes of the salicylate solution and 2 volumes of water, the portion dissolving consists of nearly pure carvol, whilst the insoluble part has the characteristics of limonene. The author has treated artificial mixtures of carvol and limonene in this manner, and has obtained results closely approximating to theory. Oil of caraway of good quality should contain at least 40 per cent. of soluble constituents.

Oil of Cedar, being composed of hydrocarbons, such as cedrene and cadinene, is insoluble in the salicylate solution.

Oil of Coriander.—The reagent dissolves the free linalool; the insoluble portion consists of limonene, pinene and linalylic esters.

Oil of Cumin.—The salicylate solution removes the cuminic aldehyde.

C. A. M.

The Action of Formaldehyde on Proteids. C. Lepierre. (*Bull. Soc. Chim.*, 1899, xxi., 729-738.)—The author has studied the action of formaldehyde on certain soluble proteid substances which do not coagulate on heating. From the results of his experiments he concludes that the action is one of dehydration and condensation, with the fixation of one or more CH_2-groups.

He finds that proto-albumoses (from muscle, fibrin, and egg albumin) are rendered completely insoluble, and the precipitate does not dissolve in hot water, in a 10 per cent. solution of sodium chloride (exclusion of hetero-albumoses), or in sodium carbonate solution. Deutero-albumoses are, in his opinion, not simple substances, but consist of a series of analogous bodies conveniently grouped together. Of these the members of higher molecular weight—*i.e.*, those approaching most nearly in composition to the proto-albumoses—become insoluble on treatment with formaldehyde, whilst those of lower molecular weight, approaching the true peptones, are converted into proto-albumoses, and only after a long-continued action of the reagent are the latter transformed into insoluble derivatives.

In like manner true peptones are said to be first converted into substances of a deutero-albumose nature, and these in turn into proto-albumoses.

The precipitates obtained from these different proteids are insoluble in cold or boiling water, but when heated for one or two hours in an autoclave at 110° C. are hydrated and rendered completely soluble, the solutions giving the characteristic reactions of the proteids from which the insoluble compounds were derived.

Contrary to the experience of Trillat, the author has found that proteids rendered insoluble are not altogether indigestible, but are dissolved by normal gastric juice, although more slowly than the untreated proteids.

With reference to the use of formaldehyde for the separation of gelatin from peptones in analytical work (ANALYST, xx., 44 and 247), it is stated that a certain amount of the peptones present are precipitated simultaneously with the gelatin, the proportion varying with the duration of the action of the formaldehyde and the nature of the peptones.

C. A. M.

Separation of the Products resulting from the Action of Pepsin on Albumin and other Proteids. J. Effront. (*Chem. Zeit.*, 1899, xxiii., 770 and 783.)—Tannin produces a precipitate in solutions of albumin, proteoses, or peptones; an excess of the reagent redissolves the peptone precipitate. If a solution of fibrin is treated with pepsin, and if to the mixture a solution of tannin, prepared exactly as below, is added, the precipitate contains nitrogen (practically) in proportion to the amount of fibrin originally taken. If the reaction between the fibrin and the pepsin is allowed to proceed for increasing periods of time, the weight of the precipitate yielded by

the tannin, and also the nitrogen in it, steadily diminishes, until, finally, no precipitation should take place. The precipitate from solutions of egg albumin with tannin contains constantly from 5·9 to 6·1 per cent. of nitrogen; it consists of 37·5 per cent. of albumin and 62·5 per cent. of tannin; extraction with water does not alter its composition; dilute alcohol removes some of the tannin, till about 45 per cent. is left. By treatment with 80 per cent. spirit, a few drops of hydrochloric acid, and ether, the albumin may be recovered pure; but the process is wasteful, and it is better to operate as follows: The precipitate is washed, dried between paper, suspended in water acidulated with a little hydrochloric acid, and dissolved by adding a sufficiency of absolute alcohol. Four or five volumes of water are introduced, followed by an excess of phosphotungstic acid. The new precipitate is collected, washed in dilute HCl, dried with paper, suspended in water, dissolved with ammonia, and thrown down with barium chloride. The filtrate is finally evaporated on the water-bath. A pure solution of albuminoids is thus produced, which, giving the xanthoprotein reaction, a precipitate with picric acid, and a deposit on saturation with zinc or ammonium sulphate shows that the original tannin-albumin mixture contained the proteoses. The filtrate from the tannin-albumin precipitate contains the peptones, and may be precipitated with phosphotungstic acid, whence treatment with barium chloride enables the peptones to be recovered pure. In their solution neither picric acid nor zinc or ammonium sulphate gives a precipitate; therefore they have been isolated.

To separate the various products of the peptonization of fibrin and albumin, two special reagents are needed. *Tannin solution:* 50 grammes of tannin are dissolved in 500 c.c. of water, 50 c.c. of normal sodium hydroxide are added, the mixture is diluted to 1 litre, and 15 c.c. of a 10 per cent. solution of tartaric acid are introduced. *Phosphotungstic acid:* A boiling 5 per cent. solution of sodium tungstate is boiled for 1 hour with 10 per cent. by volume of "medicinal" phosphoric acid;* and when cool, 15 per cent. by volume of strong sulphuric acid is added.

For analytical purposes, the original solution should contain between 5 and 10 per cent. of albumin.

(1) *Total nitrogen* is determined in 10 or 20 c.c. by the Kjeldahl process, slightly acidifying before evaporation to prevent loss of ammonia.

(2) *Total Albuminoids.*—5 or 10 c.c. of the solution are poured into 100 c.c. of the phosphotungstic acid. After twelve hours the precipitate is collected on a nitrogen-free paper, washed with semi-normal HCl, dried on the paper, and kjeldahled. In calculation the factor 6·25 is employed.

(3*a*) *Syntonins.*—From 20 or 40 c.c. of the solution, albumin is removed by coagulation under pressure. The liquid is neutralized with 1 : 10 sodium hydroxide; the precipitate brought on a small filter, washed with water, dried, and kjeldahled. More accurately, however (3*b*), the solution, freed from albumin as before, is precipitated with phosphotungstic acid (α) as it is, (β) after the insoluble products of neutralization have been filtered off. The difference gives a means of calculating the syntonins.

* The solution of phosphoric acid official in the Belgian Pharmacopœia contains 50 per cent. of H_3PO_4, and has a specific gravity of 1·35 (Squire).—ABS.

(4) *Proteoses.*—30 c.c. of the solution are freed from albumin and from syntonins with dilute soda; the volume is made up to 33 c.c. and filtered. To 11 or 22 c.c. of the filtrate are added 150 c.c. of the tannin reagent; after twelve hours the precipitate is collected on a nitrogen-free paper, washed five or six times with tannin, dried between paper, and kjeldahled.

(5) *Peptones.*—The filtrate from the proteose precipitate is mixed with 100 c.c. of phosphotungstic acid, allowed to rest for one hour; the precipitate is collected, washed with semi-normal HCl, dried between paper, and also kjeldahled.

In a liquid where peptonization has not proceeded far, saturation with zinc or ammonium sulphate and precipitation with tannin give nearly the same amounts of proteoses. The further the digestion has gone, the greater the difference between the yields, until, after seventy-two hours at 50° C., one process gives 50 per cent., the other only 33 per cent., of proteoses as the proportion of albuminoids in a solution originally containing 5 per cent. of fibrin. This anomaly is not due to the presence of peptones in the tannin precipitate, but apparently to the fact that some albumoses are thrown down by the sulphates, others not.

Phosphotungstic acid is a very untrustworthy reagent. If an almost unaltered fibrin solution, freed from albumin and syntonin, is kjeldahled, and if the phosphotungstic precipitate is also kjeldahled, the two results agree completely. If, however, digestion has proceeded far, the total nitrogen of the liquid (prepared as before) is no longer reproduced in the phosphotungstic precipitate. Experiments seem to show that "peptones" are bodies of two distinct kinds, some precipitable by the said acid, others not. The peptones themselves also appear to be attacked by pepsin. It follows that a determination of peptones from the difference between the total albuminoids and the syntonins plus proteoses does not always lead to correct figures, the error being the greater the longer the period of peptonization. Therefore, although tannin and sodium tartrate is a trustworthy reagent, and gives accurate results in the estimation of proteoses, the peptones can only be safely determined in fresh liquids; and accordingly the author's process, as a whole, is not satisfactory, if the original solution has been submitted to the action of pepsin for any considerable length of time. F. H. L.

The Use of Sozoiodol as a Reagent for Albuminous Substances in Urine. G. Guérin. (*Journ. Pharm. Chim.*, 1899, ix., 576.)—Sozoiodol, or di-iodo para-phenyl-sulphonic acid, is said to be a very sensitive reagent for abnormal albuminous substances in urine. On adding 10 to 15 drops of a 10 per cent. aqueous solution of this reagent to 8 or 10 c.c. of the filtered urine, a milky turbidity or white flocculent precipitate is produced in the presence of albuminous substances.

Alkaline urates and uric acid give no reaction; whilst albumoses, peptones, and the majority of alkaloids give a precipitate which is soluble on warming. On the other hand, nucleo-albumins give only a slight turbidity in the cold, but on heating become completely insoluble.

As the reagent undergoes alteration in the light, it should be preserved in orange-coloured bottles. C. A. M.

The Detection of Alcapton in Urine. **G. Denigès.** (*Bull. Soc. Pharm. Bordeaux*, June, 1899; through *Ann. de Chim. anal.*, 1899, iv., 312, 313.)—A urine containing alcapton is characterized by its optical inactivity (in the absence of sugar), by reducing ammoniacal silver nitrate, and by changing its colour spontaneously on exposure to the air.

The latter reaction, which is due to oxidation, has been rendered more intense by the author by treating the urine with an oxidizing agent, such as ammonium persulphate, manganese peroxide, or lead peroxide.

From 10 to 12 c.c. of the urine are mixed with 0·5 to 1·0 gramme of lead peroxide and 2 to 4 drops of a solution of sodium hydroxide, and the liquid filtered and shaken. In the absence of alcapton the filtrate remains yellow, but when that substance is present the colour varies from light to dark red.

With manganese peroxide used in the same proportion, the colour of the filtrate approaches brown, and with ammonium persulphate, of which a greater quantity is required, there is a similar coloration. C. A. M.

Analysis of Commercial Phenols. **S. B. Schryver.** (*Journ. Soc. Chem. Ind.*, 1899, xviii., 553.)—This process is based on the interaction of sodamide and bodies containing a hydroxyl group, which takes place according to the typical reaction:

$$NaNH_2 + C_6H_5OH = NaOC_6H_5 + NH_3.$$

A 200 c.c. wide-necked flask is fitted with a separating funnel, the tube of which passes to the bottom of the vessel, and an inverted condenser connected at its upper end with an absorbing vessel, and thence with an aspirator. About 1 gramme of sodamide is finely ground, washed two or three times with benzene by decantation, then introduced into the flask, and 50 or 60 c.c. of benzene (free from thiophen) added. The mixture is heated on a water-bath in a current of dry air freed from carbon dioxide for some ten minutes till the last traces of ammonia are expelled. About 20 c.c. of normal sulphuric acid are next placed in the receiver, and the phenol, dissolved in six times its weight of benzene, is brought into the funnel and allowed to drop into the flask. The funnel is rinsed with more benzene, and the current of air is maintained through the boiling liquid for one and a quarter hours. The excess of sulphuric acid is finally titrated with normal sodium carbonate and methyl orange. With phenol, cresol, and guaiacol (alone), the process gives correct results, provided (1) the apparatus and phenol are perfectly dry—sodamide acts upon water—(2) sufficient benzene is employed to hold the sodium salt in solution, (3) the benzene is free from thiophen, and (4) air is aspirated for a sufficient length of time. Toluene or xylene may replace the benzene, but in that case a sand-bath must be used instead of the water-bath.

The process is obviously not applicable to the determination of the relative proportions of more than two phenols; but it has been tested on mixtures of phenol and cresol, on wood-tar guaiacol, which is a mixture of guaiacol and creosol, on thymol in oil of thyme, and on eugenol in oil of cloves. Calling the number of c.c. of standard acid that are necessary to neutralize the ammonia given off when

1 gramme of a phenol (either simple substance or mixture) is treated with an excess of sodamide under the experimental conditions the "hydroxyl value"—which in the case of pure phenol is $\left(\frac{100}{9{\cdot}4} = \right) 10{\cdot}63$, and in that of pure cresol is $\left(\frac{100}{10{\cdot}8} = \right) 9{\cdot}26$, etc. —a table may be prepared for converting the hydroxyl value obtained when a mixture of two known phenols is operated upon directly into the relative proportion between the two ingredients, and the results calculated in this manner from the analysis of materials of the above-mentioned composition appear to be fairly satisfactory.

The method is equally available for determining the amount of water in any particular phenol, because the reaction between sodamide and water is analogous to that between the amide and a phenol. It also shows that fused sodium acetate is the best substance to remove the last traces of moisture from ordinary phenol. The process has an advantage over methods involving the use of bromine or iodine, as the results are not affected by the presence of hydrocarbons, for which reason it should be useful for estimating phenols in a large number of essential oils, etc. Sodamide acts upon ketones, amines, etc. (Titherley, *Journ. Chem. Soc. Trans.*, 1897, 460), but these bodies can be readily removed by various suitable reagents.

F. H. L.

A Critical Examination of the Methods of Quantitatively determining Salicylic Acid. **W. Fresenius and L. Grünhut.** (*Zeit. anal. Chem.*, 1899, xxxviii., 292-301.)—In their experiments on the extraction of salicylic acid by means of a solvent the authors dissolved 0·3017 gramme of pure sodium salicylate in 30 c.c. of water, and after the addition of sufficient sulphuric acid to liberate the salicylic acid, shook out the solution with four successive portions (25 c.c.) of chloroform. The united extracts were washed, evaporated at a low temperature, and the residue dried in the water-oven to constant weight. These amounts in percentages of the substance taken were: After 1 hour, 88·51; after 5 hours, 84·04; after 7 hours, 83·87; and after 9½ hours, 81·22. (Sodium salicylate contains theoretically 86·23 per cent. of salicylic acid). Thus, while the substance was still moist after 1 hour's drying, the results were too low when the drying was continued. From these results the authors conclude that it is impossible to obtain a constant weight in this way, and that only approximate determinations are possible.

Attempts were made to avoid this loss by dissolving the residue in ether before placing it in the water-bath and then employing a lower temperature. But even in this way it was not possible to determine the point at which all the solvent was expelled without any evaporation of the salicylic acid. The results thus obtained when the residue was dried at 100° C. were: After 1 hour, 85·83 per cent.; after 2 hours, 80·76 per cent.

As M. Saupe (*Pharm. Cent.*, xxxi., 314) obtained good results by drying fatty acids on the top of the water-oven instead of within it, the same process was tried with the salicylic acid, but even after the treatment with ether the residue still had an odour of chloroform, and was considerably too high.

In further experiments it was found that the drying could be effected with less

loss when a mixture of equal parts of ether and petroleum spirit was used in place of chloroform for the extraction of the salicylic acid, but that it was necessary to use the smallest possible quantity of solvent and to dry the residue on the top of the water-oven.

An iodometric method of estimating salicylic acid has been based on the observation of Messinger and Vortmann (ANALYST, xvi., 75; *Berichte*, xxii., 2312, and xxiii., 2753) that iodine acts upon phenols in alkaline solution, forming substitution products. In the case of salicylic acid the solution is heated to 50° to 60° C., and an excess of standard iodine solution added. On again warming the liquid to 50° to 60° C. a red precipitate is formed, which increases on acidifying with dilute sulphuric acid. When cold the liquid is diluted to a definite volume, and the residual iodine in an aliquot portion of the filtrate titrated with standard thiosulphate.

The authors point out that in the first communication by Messinger and Vortmann 4 atoms of iodine are said to take part in the reaction as in the equation,

$$C_6H_4\begin{matrix}OH\\COOH\end{matrix} + 4I = C_6H_3I\begin{matrix}OI\\COOH\end{matrix} + 2HI;$$

whereas in their second paper they state that the calculation must be based on the assumption that 6 atoms of iodine act upon 1 of salicylic acid, whilst Vortmann, in his *Anleitung zur Chem. Anal. organ. Stoffe*, gives the following explanation of the reaction:

$$C_6H_4\begin{matrix}OH\\COONa\end{matrix} + 3NaOH + 6I = C_6H_2I_2\begin{matrix}OI\\COONa\end{matrix} + 3NaI + 3H_2O.$$

The authors give the following results in illustration of their experience with this method, basing their calculations on the assumption that 6 atoms of iodine take part in the reaction:

	Sodium Salicylate Solution. c.c.	Normal Sodium Hydroxide Solution. c.c.	Iodine Solution added. c.c.	Acidified with Normal H_2SO_4. c.c.	Volume diluted to c.c.	Amount of Filtrate Titrated. c.c.	Thio-sulphate used. c.c.	Salicylic Acid found. Per cent.
1 ...	30	12·5	125·20	15·0	250	100	14·30	64·71
2 ...	25	12·5	126·71	15·0	250	100	16·30	74·36
3 ...	20	12·5	125·08	15·0	250	100	19·60	81·61
4 ...	20	12·5	125·02	15·0	250	100	33·89	92·70
5 ...	20	12·5	100·00	15·0	250	100	24·30	90·30

In the first three determinations the amount of sodium salicylate used was 1·0110 gramme dissolved in 100 c.c.; in the last two 1·0094 gramme in 100 c.c. In the former 1 c.c. of the iodine solution contained 0·0123215 gramme of iodine, and in the latter 0·025600 gramme of iodine.

From these results the authors conclude that this method cannot yet be relied upon to give even approximately correct results, and that the exact conditions under which it can be used as a quantitative method have yet to be determined.

The bromine method proposed by Freyer (*Chem. Zeit.*, xx., 820) has been found by them to be the most satisfactory of all the methods tried. This is based on the

facts that, on mixing a solution of salicylic acid with bromine-water in excess, a yellowish-white precipitate is formed,

$$C_6H_4\begin{matrix}OH\\COOH\end{matrix} + 8Br = C_6HBr_3{\cdot}OBr + 4HBr + CO_2;$$

and that, on adding a solution of potassium iodide, not only does the excess of bromine liberate an equivalent amount of iodine, but the tribromphenol bromide also reacts as in the equation :

$$C_6HBr_3{\cdot}OBr + 2KI = C_6HBr_3{\cdot}OK + KBr + 2I.$$

Hence, in calculating the results, only 6 atoms of bromine correspond to one of salicylic acid.

Freyer stated that an excess of about 100 per cent. of bromine was necessary, but the authors have proved that an excess of from 75 to 80 per cent. is sufficient. They have tested the method with concentrated bromine solutions, using considerable quantities of sodium salicylate, and have obtained as satisfactory results as Freyer himself. They give the following details of their method of working, in which, like Freyer and Koppeschaar, they used solutions of potassium bromate and bromide, and liberated the bromine by the addition of hydrochloric acid. The required quantity of the bromine salts solution is diluted with 300 c.c. of water, and decomposed with 30 c.c. of dilute hydrochloric acid (specific gravity 1·10). Into this mixture is introduced with continual stirring a solution of about 1 per cent. in strength of the substance under examination. A white precipitate is immediately formed, and, after this has been allowed to stand for about five minutes with occasional agitation, 30 to 40 c.c. of a 10 per cent. solution of potassium iodide are introduced and the separated iodine titrated with $\frac{N}{10}$ thiosulphate.

In the most successful results the bromide solution contained 2·5 grammes of potassium bromate, and about 10 grammes of potassium bromide in a litre of water. Twenty-five c.c. of this solution corresponded with 25·49 c.c. of thiosulphate solution, and 1 c.c. of the latter to 0·01097505 gramme of iodine or 0·00199111 gramme of salicylic acid.

The percentage of salicylic acid thus found in the same sample of sodium salicylate as used before varied in four determinations from 86·21 to 86·43 per cent.

This method is not applicable to the analysis of tabloids composed of starch and sodium salicylate. In such cases the substance should be dissolved in 90 per cent. alcohol, the solution brought to a definite volume, filtered from the undissolved starch, and an aliquot part of the filtrate used for the analysis. In a mixture of 90·91 cent. of sodium salicylate and 9·09 per cent. of starch, the authors found in this wa 89·97 per cent. of the former.

In the analysis of wines which contain sulphurous acid, aldehyde sulphurous acid, and other substances which act upon bromine, the best method of determining salicylic acid, when present, is to make the liquid alkaline, concentrate it, render it acid, and extract it with a mixture of ether and petroleum spirit. The extract thus obtained is shaken with alkaline water, which removes the salicylic acid, and this aqueous solution can then be used in the bromine method.

As regards the colorimetric method of estimating salicylic acid by means of iron

chloride, the authors state that it can only be used when the amount of salicylic acid is less than 2 milligrammes. C. A. M.

Electrolytic Recovery of Copper, Zinc, and Tin from Preserves. Hilger and L. Labaud. (*Chem. Zeit.*, 1899, xxiii., 854.)—The organic matter of the sample is best destroyed by heating with sulphuric acid, as in the Kjeldahl-Halenke method; but for the estimation of copper, treatment with 25 per cent. nitric acid is satisfactory. Zinc is deposited from an alkaline solution of its phosphate with a current of 0·2 to 0·3 ampere at about 4 volts. Tin is preferably recovered from a solution of ammonium thiostannate, or it may be thrown down with sulphuretted hydrogen, the precipitate dissolved in ammonium sulphide, and the latter liquid electrolyzed. F. H. L.

INORGANIC ANALYSIS.

Separation and Determination of Arsenic and Antimony in Ores. O. C. Beck and H. Fisher. (*School of Mines Quarterly*, 1899, xx., 372.)—This article describes an investigation into the various methods proposed for estimating arsenic and antimony in ores, which was instituted to discover the quickest and most accurate process. For the determination of the arsenic, Pearce's method—fusion of the ore with sodium carbonate and nitrate, precipitation of the arsenic with silver nitrate, and titration of the solution in nitric acid with standard ammonium thiocyanate, using ferric alum as indicator—gives slightly low results. Evaporating the ore three times to dryness with fuming nitric acid before fusing it, as above, also showed loss of arsenic (probably) by volatilization. Fusing the ore similarly in a crucible coated with fused potassium nitrate gave correct results, but the process is too long. Fischer's distillation method—decomposing the ore in hydrochloric acid and potassium chlorate, distilling the arsenic in presence of ferrous sulphate, and precipitating the distillate with sulphuretted hydrogen—is accurate, but too lengthy. Pattinson's process—solution in hydrochloric acid and chlorate, reduction of the arsenic with stannous chloride, and precipitation with sulphuretted hydrogen—is accurate and rapid, especially if the arsenious sulphide is titrated with iodine; weighing the sulphide is less satisfactory, owing to the free sulphur. For the determination of the antimony, sulphide processes come out too high; oxide processes too low, owing to mechanical loss when the sulphide is treated with nitric acid. Jannasch's method of converting the sulphide into oxide is better, but takes much time, and introduces a large amount of sulphur which must all be oxidized before ignition. Weller's process—fusion of the ore (according to Rose) with sodium hydroxide, solution of the sodium hydrogen metantimoniate in hydrochloric acid and chlorate, precipitation with sulphuretted hydrogen, re-solution of the sulphide as before, addition of potassium iodide, and titration of the excess with thiosulphate and starch—and Mohr's method—reduction of the antimonic chloride, prepared, as in Weller's process, with sulphurous acid, and titration with decinormal iodine in presence of sodium tartrate and sodium bicarbonate—gave figures which agreed closely one with another. F. H. L.

Separation of Gold from Metals containing Platinum. E. Priwoznik. (*Oesterr. Zeits. Berg. u. Hüttenw.*, 1899, xlvii., 356; through *Chem. Zeit. Rep.*, 1899, 233.)—The sample is reduced to filings, and digested with nitric acid (specific gravity, 1·199) as long as silver is dissolved. The liquid is poured off, the metal washed, and an aqua regia composed of 100 c.c. of strong hydrochloric acid, 43 c.c. of strong nitric acid, and 143 c.c. of water (which hardly attacks platinum) is added. When the surface is coated with silver chloride the acid is run off, and the deposit dissolved in weak ammonia. Six alternations between the acid and the ammonia leave pure platinum behind. The gold solutions are evaporated with hydrochloric acid till the chloride crystallizes; it is then dissolved in water, and the platinum thrown down with ammonium chloride. The gold is finally precipitated with ferrous sulphate, etc.

True alloys of gold, silver, copper, and platinum are first fused with three times their weight of lead, or preferably zinc. The melt is granulated in water, digested with sulphuric acid, and further treated as already described. F. H. L.

Separation of Copper from Tellurium. F. Stolba. (*Casopis pro prumysl chemicky*, 1899, ix., 11; through *Chem. Zeit. Rep.*, 1899, 233.)—The author's process depends on the fact that invert sugar precipitates the copper from an alkaline solution of copper below 100° C., whereas tellurium is only thrown down on boiling.

F. H. L.

Separation of Iron from Chromium, Zirconium, and Beryllium by Volatilization. F. S. Havens and A. F. Way. (*Zeits. anorg. Chem.*, 1899, xxi., 389.)—Gooch and Havens have recently shown that from a mixture of ferric oxide and alumina the iron may be completely volatilized as ferric chloride by treatment with a current of hydrochloric acid gas containing free chlorine at a temperature of 180° C. (ANALYST, 1899, xxiv., 307). The same process is available for separating iron from any of the metals mentioned in the title. In order to avoid danger of mechanical losses, the operation is best conducted at not exceeding 200° C., at which temperature 0·1 gramme of iron may be driven off in one hour, but the heat may be raised for a few minutes at the end of the process to remove the last traces of iron. The gaseous mixture is conveniently prepared by allowing sulphuric acid to drop into strong aqueous hydrochloric acid containing common salt and a little manganese dioxide. F. H. L.

Volumetric Estimation of Manganese in Permanganates by means of Arsenious Acid. C. Reichard. (*Chem. Zeit.*, 1899, xxiii., 801.)—This process depends on the fact that permanganates are reduced by an alkaline solution of arsenious acid in the cold, or by a neutral solution of the same reagent at about 50° C. The reaction proceeds according to the equation:

$$4KMnO_4 + 5As_2O_3 = 5As_2O_5 + 2K_2O + 4MnO.$$

The method possesses a double advantage: as arsenious acid can be obtained in a state of chemical purity, it serves as a means of standardizing permanganate solu-

tions, and the precipitated oxide can be treated gravimetrically to serve as a check on the titration. The liquid containing the permanganate to be analysed is mixed with an excess of standard solution of arsenious acid, heated till the manganous hydroxide falls, and then filtered; the precipitate is washed, the filtrate neutralized with hydrochloric acid, sodium bicarbonate is added, and the whole is titrated with iodine and starch. It is not even necessary to filter off the precipitate, for the manganous hydroxide can be dissolved in sulphuric acid immediately it is reduced, and the liquid titrated with a permanganate solution which has been previously standardized on arsenious acid in the presence of sulphuric acid. The examples quoted are satisfactory.

F. H. L.

Gas-Volumetric Estimation of Nitric Oxide. G. v. Knorre and K. Arndt. (*Ber.*, 1899, xxxii., 2136.)—When nitric oxide and hydrogen are led over red-hot palladium-asbestos, two reactions take place:

(*a*) $2NO + 5H_2 = 2NH_3 + 2H_2O$ (2 vols. + 5 vols. = 0 vol. + 0 vol.)
(*b*) $2NO + 2H_2 = N_2 + 2H_2O$ (2 vols. + 2 vols. = 2 vols. + 0 vol.)

The former does not take place quantitatively; therefore the amount of ammonia, or the contraction in volume, is not under control. But if the gases are passed slowly through Drehschmidt's platinum capillary tube (*Ber.*, 1888, xxi., 3245), heated to bright redness, any ammonia which may be produced is instantly decomposed, and the total reaction proceeds according to *b*. Similarly, nitrous oxide and hydrogen react as follows:

$N_2O + H_2 = N_2 + H_2O$ (1 vol. + 1 vol. = 1 vol. + 0 vol.)

The composition of a mixture containing x c.c. of nitric oxide and y c.c. of nitrous oxide can therefore be calculated thus:

$$x + y = V$$
$$1{\cdot}5\,x + y = C \text{ (contraction)}$$
$$\text{whence} \quad x = 2\,(C - V)$$

The process may be checked by measuring the hydrogen required or the nitrogen produced, so that gases composed of $NO + N_2O + N$, $NO + N$, $N_2O + N$, $N_2O + O$, etc., are all amenable to treatment.

F. H. L.

Estimation of Sulphuric Acid in Presence of Iron. G. Lunge. (*Zeits. anorg. Chem.*, 1899, xxi., 194.)—This article is mainly polemical, being a second reply to Küster and Thiel, and a discussion of the opinions advanced by those authors, by Herting, and by Meineke (*cf.* ANALYST, 1899, xxiv., 137, 164; *Zeits. anal. Chem.*, xxxvi., 209). Lunge observes that he has never recommended his process for the analysis of burnt pyrites; it was devised for the valuation of the raw ore only. He also makes the point that in Küster and Thiel's method it is difficult to tell when the proper amount of barium chloride has been added, because the suspended ferric hydroxide renders the liquid opaque; moreover, a complete extraction of the hydroxide from the barium sulphate is more troublesome and lengthy than represented by those authors.

In order to determine the relative merits of the different processes when

applied to burnt pyrites, the whole question has been reinvestigated by Lunge's assistant Bebie, who has obtained the results shown in the annexed table. The methods employed were: (A) the original Watson-Lunge process (*Zeit. angew. Chem.*, 1892, 449); (B) Meineke's process of attacking the material with sodium carbonate and potassium chlorate, precipitating the sulphur with barium; (C) solution in aqua regia, followed by Lunge's ammonia method; (D) solution in aqua regia, followed by Küster and Thiel's plan; and (E) solution in aqua regia, and reduction of the ferric chloride with zinc (Meineke).

Process.	I.	II.	III.	Mean.	Time occupied.
A	1·98	2·03	1·88	1·96	1½ hours
B	1·98	2·00	2·01	2·00	8 or 9 hours
C	1·94	1·96	1·92	1·94	,, ,,
D	1·92	1·97	1·95	1·94	,, ,,
E	1·90	1·93	1·92	1·92	6 hours

F. H. L.

[Lunge does not refer to Heidenreich's note (ANALYST, 1899, xxiv., 193); but Meineke's process (E above) appears to be identical therewith, except that the former author has written of raw pyrites, while Meineke dealt with the burnt substance. (See also following abstract.)—ABS.]

A Review of the Methods for Estimating Sulphur in Iron, Pyrites, Slags, Coal, Coke, Asphalt, Caoutchouc, and Gas-purifying Material. O. Herting. (*Chem. Zeit.*, 1899, xxiii., 768.)—It is already well known that processes for determining sulphur in iron which depend on treatment with acids and absorption of the sulphuretted hydrogen are incorrect, for part of the sulphur is evolved as an organic compound (methylic sulphide), and this portion is not oxidized by bromine and hydrochloric acid or by hydrogen peroxide. Phillips, however, has proved that ignition in presence of hydrogen and carbon dioxide is quite efficient to convert the organic sulphur into sulphuretted hydrogen; and, based on this observation, Campredon has proposed absorbing the products of combustion in zinc acetate acidified with acetic acid, titrating the zinc sulphide with normal iodine and thiosulphate; while Schulte prefers to use acid cadmium acetate, converting the cadmium sulphide into copper sulphide, and weighing the latter in the form of oxide. Both methods are satisfactory; Campredon's is somewhat quicker in operation. For the determination of total sulphur in iron, Blair has described a process (ANALYST, 1897, xxii., 279) which is very suitable for occasional employment, and where great rapidity is not essential. For routine purposes the author is engaged in elaborating a colorimetric method with cadmium in Wiborgh's apparatus, details of which will follow. It is not possible by carrying out these absorption processes without the intermediate combustion tube, and calculating from the sulphuretted hydrogen so evolved, to ascertain the amount of "organic sulphur" in iron. Schulte's results have shown that the proportion of "organic sulphur" varies within very wide limits even in the same grades of metal.

To determine sulphur in raw pyrites Lunge's process is quite properly employed. Küster and Thiel's modification (ANALYST, 1899, xxiv., 137), according to Herting, is

"worthy of notice." For practical purposes the plan outlined in Heidenreich's preliminary note (ANALYST, 1899, xxiv., 193) is more serviceable; but the beaker should be kept well covered and in the dark while the barium sulphate is settling. The method has been improved by Lehnardt, who adopts stannous chloride instead of metallic zinc as the reducing agent, thus saving considerable time. For burnt pyrites fusion processes with alkali carbonates and nitre are the best. Slags from the puddling furnace should be treated like iron ores; blast-furnace slags by solution in bromine and hydrochloric acid. In the latter case the silica should be removed and the sulphuric acid precipitated directly, for the percentage of iron in such materials is generally low. The sulphur in blast-furnace slags may be estimated volumetrically by rubbing down 0·5 gramme in an agate mortar, adding 150 c.c. of warm water and a little starch, then introducing an excess of decinormal iodine and 20 c.c. of hydrochloric acid, agitating for one minute, and titrating with thiosulphate.

For coal, coke, and asphalt Eschka's process as improved by Hundeshagen is the most suitable, the absorbing material being magnesium oxide mixed with equal parts of sodium and potassium carbonate, or with potassium carbonate only. For indiarubber Unger's method is to be recommended: 0·5 gramme in very small pieces is carefully ignited with 12 grammes of copper oxide and 2 grammes of sodium carbonate. The melt is taken up in aqua regia, evaporated to remove the antimony, filtered and precipitated with barium chloride; the barium sulphate is fused with sodium carbonate, and the sulphuric acid thrown down a second time.

Estimation of sulphur in spent oxide by extraction with carbon disulphide yields results which are too high owing to the presence of tar, etc. The substance should be dried at 75° C., for six hours, ground till it will pass through a sieve of 400 meshes per square centimetre, then exposed to air for one hour. One gramme is next extracted with carbon disulphide, and the dried extract is oxidized with 25 c.c. of warm 1·19 hydrochloric acid and a few crystals of potassium chlorate; the excess of chlorine is driven off, and the sulphuric acid determined as usual. F. H. L.

Determination of Oxygen in Water. F. Zetsche. (*Zeit. für Untersuch. der Nahr. und Genussmittel*, ii., 696, 697.)—The author has had considerable experience with Winkler's method for the open-air determination of oxygen in water, and regards it as the most practicable of any method devised for the purpose. His mode of working is as follows:

Apparatus and Chemicals Required.—(*a*) A number of glass-stoppered flasks (according to the number of samples to be taken), measuring from 250 to 300 c.c. capacity, and each containing ten to fifteen small porcelain balls. (*b*) A saturated solution of manganous sulphate (say 50 c.c.). (*c*) A solution containing 48 grammes of sodium hydrate and 15 grammes of potassium iodide in 100 c.c. of water. (*d*) 500 c.c. of concentrated hydrochloric acid. (*e*) Three pipettes, to deliver 1, 2, and 5 c.c. respectively. The capacity of each of the flasks must be accurately determined after the introduction of the balls.

Procedure.—The flasks are filled to overflowing with the water to be examined, and 1 c.c. of the manganous sulphate solution and 2 c.c. of the alkaline potassium

iodide solution are run in to the bottom of each by means of pipettes. The flasks are then closed and briskly shaken, the movement of the balls insuring complete mixing. When the brown precipitate produced has settled, the flasks are opened and 5 c.c. of hydrochloric acid run in (also to the bottom of the flasks). The flasks are then closed and again shaken, and as soon as the precipitate in each has dissolved, the liquid is transferred to a beaker, and the liberated iodine titrated with $\frac{N}{100}$ sodium thiosulphate solution. In calculating the results, allowance must of course be made for the 3 c.c. of reagents added. H. H. B. S.

The Cleaning of Crucibles. F. Wirthle. (*Chem. Zeit.*, 1899, xxiii., 803.)—Platinum crucibles in which vegetable substances have been incinerated are far more easily cleansed by melting borax in them than by the use of the favourite potassium bisulphate. Porcelain crucibles soiled with silver chloride, lead sulphate, stannic oxide, etc., are readily cleansed by filling them with acid and adding a fragment of zinc; after a short time the reduced metals can be completely removed. The author suggests that this electrolytic method of decomposing insoluble precipitates should perhaps be quicker in qualitative analysis than the usual plan of fusing them with various fluxes. F. H. L.

REVIEW.

THE CHEMISTRY OF ESSENTIAL OILS AND ARTIFICIAL PERFUMES. By ERNEST J. PARRY. London: Scott, Greenwood and Co. Price 12s. 6d. net.

Owing to the vigorous manner in which the study of the essential oils has been prosecuted during recent years, and to the enormous number of valuable, and in many cases brilliant, researches which have during that short period been brought to a successful conclusion, we have fully arrived at the point at which the services of a painstaking and careful gleaner become extremely desirable, and this service Mr. Parry has rendered in compiling and issuing to the world the volume under review.

Seeing that no work covering similar ground exists in the English tongue, British chemists will be quite ready to confirm the author in his belief that its appearance demands no apology.

Chapter II. deals with the properties of those compounds which are found in essential oils, but does not treat the subject as exhaustively as one could have wished, seeing that a work of this character must necessarily appeal rather to the chemical specialist than to the general chemist. It is with the analytical portion of the book that I am now chiefly concerned, but I may remark in passing that cymene is para-methyl-iso-propyl-benzene, and not the normal propyl compound, and that thymol and carvacrol are likewise iso-propyl derivatives. Further, I do not notice any reference to the open chain hydro-carbons which have been found in some oils, and which are of considerable interest.

When one remembers the very large trade interests involved, and the ease with which the sophistication of valuable oils could until quite recently (and in some cases

can still) be practised, the need of analytical processes capable of detecting, and so of preventing, these sophistications becomes at once apparent.

Moreover, it has to be borne in mind that most essential oils vary in composition within certain limits, and that the most variable constituent is often that upon which the value of the oil chiefly depends.

Analytical processes are therefore necessary for the evaluation of these oils as well as for the detection of adulteration.

Mr. Parry has devoted one chapter to a discussion of certain methods, which are generally applicable to essential oils, dealing with the processes for the estimation of certain special constituents in later sections of the book, an arrangement which certainly makes for convenience.

In the general section some details in regard to the determinations of specific gravities are given, the use of a bottle being recommended for this purpose, a sprengel tube being only employed when the volume of oil at the analyst's disposal is too small for the former method. But surely it is in the case of liquids, like the majority of essential oils, having high co-efficients of expansion and great mobility, that the superiority of the sprengel tube over the bottle is so evident. We are next treated to the inevitable dissertation on the polarimeter, its construction and use. Both these matters might well have been omitted, seeing that every chemist concerned in the analytical examination of essential oils might well be supposed to be conversant with them. On the other hand, many analysts would doubtless have welcomed some information in regard to the simplest and most convenient apparatus for distillation under reduced pressure. It is the process which has to be carried out prior to the scientific investigation of practically all essential oils, and one that can often be resorted to with great advantage in their analytical examination. A few hints in regard to forms of receivers, distillation in an atmosphere of carbonic acid gas, and other points, might have been of service to many chemists. In this section no mention is made of the "methyl number," a useful determination in some cases.

The special analytical processes are clearly and carefully described, and the book will undoubtedly prove of considerable value to all chemists who are engaged in the examination of these oils.

Mr. Parry has evidently spared no pains in selecting from the enormous masses of recorded results those to which the greatest amount of probability attaches, and he has given numerous references to the original papers when they are likely to be of value to the reader. The book is well printed, and possesses a good index.

In many places, however, the wording is a little loose. One would like to know for example what is meant by the expression "excretionary functions" as applied to essential oils. Presumably it should refer to the cell. The words "under reduced pressure" should be substituted for "in vacuo," in speaking of distillation, and "rotatory" for "rotary."

There are many little blemishes of a literary character which, whilst in no way detracting from the real value of the book, yet tend to diminish the pleasure with which one reads it. These would doubtless disappear in a future edition.

A. C. C.

THE ANALYST.

FEBRUARY, 1900.

OBITUARY NOTICE.

THE LATE MR. GEORGE JARMAIN.

We regret to have to chronicle the death, at the age of seventy-three years, of an old member of our Society, Mr. G. Jarmain, F.I.C., F.C.S., which took place at Huddersfield on January 16. Mr. Jarmain held the post of Public Analyst to the Huddersfield Corporation since the institution of that office. He was a native of York, and received his education at the Training College there. He first adopted the scholastic profession, but relinquished it for the study, and subsequently for the practice, of chemistry. He lectured at the Huddersfield Technical College for about forty years, resigning his post there only five or six years ago. His name is associated with several elementary works on chemistry, including his well-known and, for beginners, valuable little book on "Qualitative Analysis."

PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS.

The annual general meeting of the Society was held on Wednesday, January 24, in the Chemical Society's Rooms, Burlington House. The President (Mr. W. W. Fisher, M.A.) occupied the chair.

The minutes of the previous meeting were read and confirmed.

A certificate of proposal for election to membership in favour of Mr. B. W. J. Warren, A.I.C., was read for the second time; and certificates in favour of Messrs. Norman Parr Booth, assistant to Mr. Wm. Chattaway, Apothecaries' Hall, London; John Macallan, A.R.C.S.I., F.I.C., chief assistant to Sir Charles A. Cameron, Municipal Buildings, Dublin; and C. T. Tyrer, Works Manager and Analytical Chemist, Stirling Chemical Works, Stratford, were read for the first time.

Messrs. J. A. Foster, J. B. P. Harrison and D. Lloyd Howard were elected members of the Society.

The Hon. Treasurer (Mr. E. W. Voelcker), in presenting the accounts of the Society for the past year, said that the balance of receipts over expenditure during 1899 was considerably less than had been the case in the previous year. In 1898 he successful, with only one exception, in collecting all the subscriptions that

were due, including nine outstanding from earlier years. Of the 1899 subscriptions nine remained in arrear at the end of the year, while there had only been one outstanding subscription to collect, and the balance of income for 1899 had been affected accordingly. The expenditure during the year had been carefully kept down. The cost of the ANALYST was slightly less than in the previous years. The expenses of meetings were somewhat higher, owing to the fact that one more general meeting and three more council meetings had been held. In petty disbursements, postage, and expenses of a similar class, a considerable saving was shown, while in the case of printing and stationery there was an increase of about £3. A somewhat unusual item of expenditure appeared, namely, law charges, which had been incurred for the benefit of a member whose interests had been in danger of being prejudicially affected by the action of a certain County Council. In the early part of 1899 the Society's investments had been increased by the purchase of £100 2½ per cent. annuities. The securities held by the Society comprised £300 Consols and £200 2½ per cent. annuities, as against only £150 Consols at the end of 1894, an increase which, made as it had been in the short space of five years, might be regarded with considerable satisfaction.

On the motion of Mr. G. EMBREY, seconded by Mr. P. A. E. RICHARDS, the accounts were unanimously adopted.

Votes of thanks to the Hon. Treasurer and Auditors were unanimously passed.

The President then delivered his annual address.

A vote of thanks to the President for his address was proposed by Mr. HEHNER, with the request that he would allow it to be published. This was seconded by Dr. DYER, and carried unanimously.

The PRESIDENT, having responded, announced that the list of officers and members of Council nominated for the ensuing year had been without any exception accepted, and expressed his thanks to the Society for again placing him in the position of President. He felt that in that position he owed a great deal to the officers of the Society, and especially to the Hon. Secretaries as well as to the senior members of the Council.

A vote of thanks was unanimously passed to the President and Council of the Chemical Society for permission to use their rooms during the past year.

OFFICERS AND MEMBERS OF COUNCIL FOR 1900.

President.—W. W. Fisher, M.A.

Past-Presidents.—M. A. Adams, F.R.C.S., Alfred H. Allen, Sir Chas. A. Cameron, C.B., M.D., F.R.C.S., A. Dupré, Ph.D., F.R.S., Bernard Dyer, D.Sc., Otto Hehner, Alfred Hill, M.D., J. Muter, M.A., Ph.D., Thos. Stevenson. M.D., F.R.C.P.

Vice-Presidents.—Charles E. Cassal, E. W. T. Jones, S. Rideal, D.Sc.

Hon. Treasurer.—E. W. Voelcker, A.R.S.M.

Hon. Secretaries.—E. J. Bevan, Alfred C. Chapman.

Other Members of Council.—L. Archbutt, A. Ashby, M.B., F.R.C.S., G. Embrey, J. H. B. Jenkins, Arthur R. Ling, C. A. Mitchell, B.A., James Nimmo, Thomas A. Pooley, B.Sc., P. A. E. Richards, H. Droop Richmond, R. T. Thomson, Rowland Williams.

ANNUAL DINNER OF THE SOCIETY OF PUBLIC ANALYSTS.

THE annual dinner was held at the Criterion Restaurant after the annual meeting, under the chairmanship of the President (Mr. W. W. Fisher, M.A.). There were present over seventy members and guests, among the latter being Mr. R. T. Prowse, C.B., Secretary H.M. Customs Establishment; Prof. T. E. Thorpe, F.R.S., President of the Chemical Society; Dr. Thos. Stevenson, F.R.C.P., President of the Institute of Chemistry; Dr. Stocker, Master of the Society of Apothecaries; Mr. W. Martindale, President of the Pharmaceutical Society; Mr. F. M. Mercer, Master of the Wheelwrights' Company; Mr. C. E. Groves, F.R.S.; Prof. Frank Clowes, D.Sc.; Dr. Henry E. Armstrong, F.R.S.; Dr. F. D. Chattaway; Mr. E. Rigg, M.A.; Mr. Edward Holmes; Mr. E. M. Holmes, F.L.S.; Mr. W. J. Fisher; Mr. W. P. Squire; and Mr. Aubrey W. Rake.

The following toasts were proposed: "The Queen," by the President; "The Navy, Army and Reserve Forces," by the President, responded to by Major Cassal; "The Institute of Chemistry," by Mr. Otto Hehner, responded to by Dr. Stevenson; "Kindred Societies," by Dr. Armstrong, responded to by Prof. Thorpe; "The Society of Public Analysts," by Prof. Frank Clowes, responded to by the President; "The Guests," by Dr. Bernard Dyer, and responded to by Mr. R. T. Prowse, C.B.

ON THE DETERMINATION OF THE IODINE VALUE.

BY DR. J. J. A. WIJS.

(*Read at the Meeting, December* 6, 1899.)

I READ with great pleasure the paper by Dr. Lewkowitsch on this subject recorded in the ANALYST, p. 257, and am desirous of making the following observations on the subject-matter of it:

I. *Iodine Values found for Allyl Alcohol.*—I fully agree with Dr. Lewkowitsch that allyl alcohol is not a substance specially suitable for deciding the respective values of the different methods for determining iodine values. The rapidity with

which the addition process takes place in allyl alcohol being less than that of the oil which has the smallest rapidity (linseed-oil), allyl alcohol demands a greater additive force on the part of the liquid than is necessary in its ordinary application.

From some experiments published in my first paper (*Zeits. f. Anal. Chem.*, 1898, 277), Dr. Lewkowitsch calculates for allyl alcohol the iodine value 392. These experiments are, however, unsuitable for this purpose. The whole series was made merely with a view to determining the splitting off of acid from the ICl-addition product, and in order to have this splitting-off process as uncomplicated as possible, and not affected by the formation of acid which always takes place when a Hübl solution is allowed to stand for any length of time, the duration of the reaction was kept very short (quarter to half-an-hour). The iodine values calculated from these experiments are therefore much too low, particularly for substances such as allyl alcohol, linseed oil, or cholesterol, which possess a small rapidity of addition.

Dr. Lewkowitsch obtained with ICl-acetic acid liquid for a sample of allyl alcohol iodine values of 419-420, and with the Hübl method 421-422, whereas I found with the same sample with ICl-acetic acid liquid 429, and for another sample by Hübl's method 425. The difference between the results obtained with the same method depends, as is evident from a letter I received from Dr. Lewkowitsch, upon a different method of working, *i.e.*, having a different excess of iodine present. I always used an excess of about 75 per cent. of the quantity originally added, whereas Dr. Lewkowitsch writes that he also in every case employed an excess of 75 per cent., and in most cases considerably more, approximating to 100 per cent. It is evident that this can only have been 75 to 100 per cent. of the quantity of halogen absorbed, *i.e.*, only 43 to 50 per cent. of the quantity originally added. It is, therefore, not at all a matter of surprise that Dr. Lewkowitsch obtained lower iodine values than mine.

II. *Effect of Time on the ICl-acetic Acid Liquid.*—Dr. Lewkowitsch finds that a solution of iodine chloride in 95 per cent. acetic acid is much more liable to alteration (decrease) than a solution in an acid of 99 per cent. When the acid is pure and especially free from oxidizable matters, there is, theoretically, no possibility of any decrease. Dr. Lewkowitsch found that a solution in 95 per cent. acid, after sixty-four hours, had decreased 4 per cent. of its value; whereas I had stated (*Berichte d. deutsch. Chem. Ges.*, xxxi., 750) the decrease to be 0·3 per cent. in ninety-six hours. I can find no other explanation for this difference than the assumption that Dr. Lewkowitsch's 95 per cent. acid was not so pure as mine.

III. In the third place, Dr. Lewkowitsch writes that the iodine determinations made with Hübl solution lead to correct results provided that the Hübl method is applied in a proper manner, and further on he says that the solution should not be allowed to act longer than six or seven hours on the fat under examination. I must add here that this limitation to seven hours only bears reference to those cases in which, in the first place, fresh (*e.g.*, sixteen-hours-old) iodine solution is used; and, secondly, where an excess of about 75 per cent. of the added iodine is employed. When treating oils of a high iodine value (*e.g.*, linseed oil) with a Hübl solution that is not fairly fresh, say about five days old, twenty-four hours are required.

It is clear that what I call a proper application of the Hübl method is but little

different from the usual one carried out in the case of oils with a low iodine value; whereas in the case of oils with a high iodine value the difference is greater.

I was therefore convinced that the iodine value of linseed-oil especially required a revision, and I made one (*Revue ü. d. Fett- und Oelindustrie*, 1899, 29). This paper not having appeared in the ANALYST in abstract form, I give a short account of it.

I determined the iodine values of eighty-six samples of guaranteed pure linseed-oil, all from seeds of known origin; sixty-nine were pressed from unmixed seeds, and seventeen from mixed. The results were as follows:

	Iodine Values.	
Dutch linseed oils	From 191·5 to 201·8	
North Russian (Baltic) oils ...	,, 192·1 ,, 200·0	(only one had a value 188·5)
South Russian oils	176·3 186·3	
Indian oils ...	182·2 187·5	
La Plata oils ...	174·7 182·7	
North American oils	177·8 188·5	

The more rapidly-drying varieties (Dutch and North Russian), which are only used for paints and varnishes, have higher iodine values than those used solely for soap-making. I also determined the specific gravity at 15° C. of twenty-four of these samples, and found the following relation between this constant and the iodine value:

	Iodine Values.	Specific Gravity.
One sample	Higher than 200	... 0·9352
Mean of four samples ...	... Between 195·0 and 200·1 ...	... 0·9339
,, ,, ,, ...	... ,, 190·0 ,, 195·1 ...	... 0·9329
,, ,, ,, ...	... ,, 185·0 ,, 190·1 ...	... 0·9322
,, six ,, ...	... ,, 180·0 ,, 185·1 ...	... 0·9317
,, five ,, ...	Lower than 180·1	... 0·9310

DISCUSSION.

Dr. LEWKOWITSCH said that although Dr. Wijs and himself were more or less in agreement on this subject, there were one or two points raised in the paper which seemed to require correcting or amplifying. He was very glad that after all Dr. Wijs agreed with him in thinking that allyl alcohol was not a suitable substance to decide the question at issue. It had, in fact, been introduced by a third party, and it was this that had led him (the speaker) to calculate, from one of Dr. Wijs' former papers, the number 392. He quite accepted the author's remarks on this point. Dr. Wijs thought that he could explain the divergence in the iodine numbers of the same sample of allyl alcohol examined by both (viz.: Wijs, 426; Lewkowitsch, 420), by the fact that different excesses of the reagent had been used. There was, indeed, some misunderstanding as to the excess to be employed. Dr. Wijs considered that if from 100 parts of iodine added 25 were absorbed, an excess of 75 per cent. was used, whereas he (the speaker) should call that an excess of 300 per cent. However, he had started a number of experiments for the purpose of settling this point. The following figures showed that the variations between different observations were sometimes greater than those due to differences of excess, and it would seem that it did not really much matter whether one worked with an excess—in Wijs' sense—of

75 per cent. or 43 per cent. (or, as he would call it, 300 per cent. and 100 per cent. respectively):

Substance.	Iodine Value.	Excess of Iodine in W.'s sense. Per cent.	Excess of Iodine in L.'s sense. Per cent.
Allyl alcohol	413·2	73·7	281·2
,, ,, ...	416·0	52·8	89·3
,, ,, ...	416·0	52·8	89·3
Linseed oil	179·8	77·6	347
,, ,, ...	180 9	76·0	315
,, ,, ...	179·3	57·2	133
,, ,, ...	178·6	52·5	110
Candle-nut oil	163·4	73·6	278
... ...	163·4	72·99	270·3
... ...	162·1	53·4	115·0
.. ..	159·1	49·5	97·9
... ...	163·9	77·5	308·9
... ...	163·9	77·5	308·9
... ...	162·1	51·6	106·8
... ...	162·1	51·6	106·8
Oleic acid	82·9	76·1	319·9
,, ,, ...	83·25	73·9	283·2
,, ,, ...	82·81	52·7	115·5
,, ,, ...	82·82	50·0	100·4

As a rule, the excess he used was not calculated beforehand, but left more or less to chance, and varied generally between, say, 100 per cent. and 150 per cent. (in his own sense). But even making the largest possible allowance, the difference of about 1 per cent. in the value of allyl alcohol did not explain the difference of Dr. Wijs' own figures for allyl alcohol. He thought, therefore, that the matter might fairly be allowed to drop there, accepting the conclusion that it did not really matter whether the excess was 300 per cent. or half of that, because, according to the law of mass action, secondary reactions were bound to occur with the possible formation of substitution products, thus probably introducing greater errors than those due to differences in the excess. The temperature variations had only a very slight influence, as Dr. Wijs himself had shown. The explanation Dr. Wijs had given for the somewhat rapid decomposition of the speaker's solution, prepared with 95 per cent. acetic acid—viz., that the acid had been impure—did not hold good, it having been prepared from pure glacial acetic acid, it was the same sample from which the 100 per cent. glacial acid solutions were made later on. With regard to the figures given by Dr. Wijs for the various linseed oils, these numbers were very valuable as far as they went, but they ought not to be taken as indicating the order in which the oils derived from the various countries ranked in quality (or purity). He himself had examined some Indian linseed oils, the iodine value of which came up to 193, tested by the old Hübl process, allowing the solution to act from four to six hours. The specific gravity table showed the regularity one would expect, as the higher the iodine value, *i.e.*, the larger the quantity of linolic and linolenic acid present, the higher the specific gravity must be, since the specific gravities of linolic and linolenic acids were higher than

that of oleic acid. But it would not be permissible to allow the rule shown by the table to pass into literature as a general one.

Mr. A. MARSHALL said that, in his experience, solution made even with perfectly pure acetic acid, although it lost very little strength during the first few days, showed on keeping for some months a considerable decrease in strength, and became much darker, probably owing to chlorination of the acetic acid and reconversion of the iodine chloride into iodine.

Dr. LEWKOWITSCH said that in Dr. Wijs' process long hours of standing were not permissible at all. The author, in his fourth paper, had proved pretty accurately the occurrence of side reactions after a certain time, which affected the result in a reverse direction. This was well known to every observer, for if after titration of the iodine the solution were allowed to stand it became blue again. One of the chief points in Wijs' work was in showing that the importance which had been attached to a standard time for exposure was well founded, and that it was necessary to adhere strictly to a certain period, sufficiently long to insure the reaction coming to an end. He (Dr. Lewkowitsch) had been able to show that Hübl's method gave identical results with that of Wijs', but in the case of the latter the solution would keep for at least half a year, so that a blank determination was not absolutely necessary, and actually less time was required for a test than for a determination of the saponification value. It was so easy in preparing the Wijs' solution to hit exactly the point when the titer was doubled that there seemed to be no necessity for having an excess of either chlorine or iodine. He considered that a point had been reached when the discussions on, and the endless modifications of, the Hübl process might with advantage cease.

ON A METHOD FOR DISTINGUISHING BETWEEN HOPS AND QUASSIA.

BY ALFRED C. CHAPMAN, F.I.C.

(*Read at the Meeting, December* 6, 1899.)

IN the course of an investigation of the resinous constituents of hop-flowers, the results of which have been published elsewhere, I observed that these substances, when extracted from the hops by means of ether, yielded a somewhat considerable quantity of valeric acid on oxidation with an alkaline solution of potassium permanganate.

It occurred to me at the time that this fact might serve as the basis of a method for distinguishing between hops and quassia in fermented and other beverages, and the following experiments which I have since made show that such is the case.

In the first place, quassia-chips themselves were extracted with dry ether, and the small quantity of extract so obtained was submitted to the action of alkaline permanganate solution, but no valeric acid was obtained.

In order to arrive at the best conditions for the application of this method, fermented liquids of various kinds, bittered with hops on the one hand and with quassia on the other, were prepared in the laboratory in the following manner:

Cane-sugar solutions and malt-worts were prepared, containing in some cases 5 per cent., and in others 10 per cent., of dry solids. Each of these was divided into two portions, hops being added to the one at the rate of 10 grammes per 500 c.c. of the solution, whilst 2·5 grammes of quassia chips were added to the other. For equal weights, the quassia wood has roughly four times the bittering power of good strong hops, and if used to replace the latter, the quantity to be employed would probably be calculated on this basis. Both solutions were then boiled gently in covered beakers for one hour. At the end of this time they were filtered, yeast was added after cooling, and the solutions were allowed to ferment for several days.

The "artificial beverages" so prepared were then submitted to the following treatment: 500 c.c. were evaporated on a water-bath in a porcelain basin, fine recently-ignited sand being added towards the end of the operation, and the mass constantly stirred with a steel spatula to prevent it from adhering strongly to the surface of the dish. The residue was then transferred to an air-oven, and when quite dry was finely powdered in a mortar. This powder was next digested for some time with ether in a stoppered bottle, and the ethereal extract filtered into a flask, the ether being recovered by distillation. The flask was placed on a water-bath to drive off the last traces of ether, and the small quantity of residual matter oxidized by the careful addition of an alkaline solution of potassium permanganate, containing 40 grammes of parmanganate and 10 grammes of caustic potash per litre. This solution should be added in very small quantities at a time, the flask being vigorously shaken, and if necessary warmed. When the permanganate ceased to be readily reduced, a few drops of a hot solution of oxalic acid were added to complete the reduction, and the colourless liquid was filtered from the oxides of manganese into a glass dish, in which it was evaporated to dryness. The dry residue was then acidified with dilute sulphuric acid, when the odour of valeric acid in the case of the hop-bittered liquid became at once apparent, being rather accentuated by the carbon dioxide liberated at the same time from the potassium carbonate formed during the oxidation.

I may say that the smell observed is not that of pure valeric acid, but of valeric acid plus some other odorous compound, which serves to render it more characteristic.

In the case of the quassia experiment the liberated acid is chiefly acetic, with, of course, carbon dioxide, and only in a few cases was there any marked odour, which, however, was altogether different from that obtained from hops.

In the above experiments old hops responded as readily to this test as new hops.

Experiments were next made with beer itself. One litre of the beer was evaporated with sand, and the residue from the ethereal extraction treated as described above. In one or two samples of mild ale the indication was uncertain, but in all the samples of bitter beer and in the majority of the mild ales and porters examined this valeric smell was unmistakable.

Camomile extract behaves in a similar manner to hops, but chiretta yields no valeric acid.

This method is readily applicable to the examination of hop-bitter preparations (of a medicinal character), hop extracts, and similar products. It is also capable of furnishing additional evidence in the case of fermented beverages which have been

examined according to the systematic schemes in vogue, and which have yielded results of an uncertain nature.

In applying this method, it is important not to carry the oxidation of the ether residue too far. At first the permanganate solution is reduced readily, the mixture becoming warm, but towards the end it is usually necessary to heat slightly. With a very little experience there is no difficulty in arriving at the proper conditions, which cannot be definitely laid down, since the matter extracted by the ether varies both in character and quantity, and it must therefore be left to the operator to decide when the oxidation has been carried sufficiently far.

ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

FOODS AND DRUGS ANALYSIS.

The Detection of Formalin in Milk. A. Leys. (*Ann. de Chim. anal.*, 1899, iv., 338-342.)—The author describes the tests used in the Municipal Laboratory in Paris. The one principally relied upon is that of Jorissen (ANALYST, xxii., 282), in which 25 c.c. of the milk are shaken with 10 c.c. of a solution of phloroglucinol (1 gramme per litre), and subsequently with 5 to 10 c.c. of a solution of potassium hydroxide (1 of the ordinary laboratory solution with 2 of water).

It has been found, however, that in the proportion in which formalin is frequently met with in milk in Paris (1 : 10,000) there is a possibility of the colour being mistaken for that given by the potassium hydroxide alone with milk which has previously been boiled or artificially coloured with annatto, turmeric, or chrysoin yellow, and to eliminate these chances of error the following preliminary tests are made :

Ten c.c. of the milk are shaken with 20 c.c. of Adams' ammoniacal mixture of alcohol and ether. If after a few seconds the lower layer is completely opaque instead of being semi-transparent, the milk is regarded as having been boiled, thus altering the nature of the casein. When the lower layer is of a greenish tint and slightly fluorescent, annatto is indicated, and may be identified as described by the author in former papers (ANALYST, xxiii., 174 and 230). A red coloration of the lower layer points to the presence of turmeric, whilst chrysoin causes the upper layer to be golden yellow, and the milk itself gives a reddish-brown tint on treatment with potassium hydroxide.

When, in the absence of these substances, formalin has been detected by Jorissen's reagent, confirmatory evidence is obtained by distilling the milk and testing the distillate with Gayon's reagent (0·1 per cent. aqueous solution of fuchsin, 1,000 c.c. ; sodium bisulphite solution, 30° Bé., 10 c.c.; and concentrated hydrochloric acid, 10 c.c.). The milk itself is also tested by Denigés' method (ANALYST, xxi., 285).

C. A. M.

Chemical Composition of Butter Fat. C. A. Browne, jun. (*Journ. Amer. Chem. Soc.*, vol. xxi. [10], pp. 807-827.)—The quantities of mixed acids and corresponding triglycerides determined in the butter examined are:

	Acid. Per Cent.	Triglyceride. Per Cent.
Dioxystearic	1·00	1·04
Oleic	32·50	33·95
Stearic	1·83	1·91
Palmitic	38·61	40·51
Myristic	9·89	10·44
Lauric	2·57	2·73
Capric	0·32	0·34
Caprylic	0·49	0·53
Caproic	2·09	2·32
Butyric	5·45	6·23
	94·75	100·00

The unsaponifiable matter, amounting to only about 0·1 per cent., was not further investigated.

An elementary analysis of the fat gave results very closely according with the theoretical calculation from the above table, the values being:

	C. Per Cent.	H. Per Cent.	O. Per Cent.
Actual	75·17	11·72	13·11
Theoretical	74·86	11·71	13·43

C. S.

New Method of Estimating Starch. D. Crispo. (*Ann. de Chim. anal.*, 1899, iv., 289, 290.)—This is a rapid process, based on a polarimetric examination of an alkaline solution of the starch; a 10·1732 per cent. solution of starch examined in a 200 mm. tube, read at 100 Soleil-Ventzke-Scheibler divisions. 3·391 grammes of the sample are triturated with a little water, 50 c.c. of a 6 per cent. solution of potassium hydroxide added, and the liquid, after being diluted to about 140 c.c., kept for an hour on a boiling water-bath with constant agitation. When cold it is made up to 200 c.c., filtered, and examined with the polarimeter in a 200 mm. tube. The number of divisions, multiplied by 6, gives the percentage of anhydrous starch.

The following results were thus obtained in the analysis of specimens of different commercial starches:

	Potato Starch.			Maize Starch.	Rice Starch.
Starch by polarization ...	81·3	80·7	81·9	85·45	85·05
Ash	0·3	0·3	0·3	0·31	0·75
Proteid substances	0·2	0·2	0·2	0·38	0·50
Water	17·9	17·9	17·9	14·42	14·56
	99·7	99·1	100·3	100·56	100·86

It has not yet been determined whether this method can be used for the determination of starch in cereals, but the lævorotatory products formed by the

action of the potassium hydroxide on the gluten and other nitrogenous substances in the grain would probably be a source of error. C. A. M.

Estimation of Starch in Yeast. D. Crispo. (*Ann. de Chim. anal.*, 1899, iv., 220, 291.)—This is an application of the author's general polarimetric method (see preceding abstract). Fifty grammes of the yeast, which must be left until it has become soft, brown, and viscous, are stirred up in about 2 litres of water, and allowed to settle for from ten to twenty minutes. The supernatant liquid is siphoned off, and the deposit again washed four or five times in the same way, until the washings are almost clear. Finally the deposit is transferred to a graduated flask, where it is mixed with water in the proportion of about 100 c.c. to each 1 to 2 grammes of starch. A solution of potassium hydroxide is then introduced, with continual agitation, so that for each gramme of starch there is 1 gramme of alkali. The flask is filled to about three-fourths with water, heated on the water-bath for an hour, and the resulting solution examined with the S.-V.-S. polarimeter in a 200 mm. tube.

The number of divisions, multiplied by 0·10173, gives the percentage of anhydrous starch in 100 c.c. of the solution. The amount may be calculated on the moist yeast on the assumption that the latter contains on the average 18 per cent. of water. The mean correction for the starch removed during the washing has been determined experimentally by the author to be 11 per cent.

The following analyses of test mixtures of yeast and starch were made by five different chemists, using this method :

Starch added per cent.	13·8	5·66	1·96	3·85	6·00	4·00	2·00	10·0	5·0
,, found ,,	13·0	6·4	2·29	4·35	6·50	4·00	2·02	10·32	4·97

C. A. M.

The Detection of Sawdust in Flour. G. A. Le Roy. (*Ann. de Chim. anal.*, 1899, iv., 212, 221.)—After making experiments with various reagents used for the detection of woody fibre in paper, the author has obtained the most satisfactory results with an acid solution of phloroglucinol.

This is prepared by mixing 1 gramme of phloroglucinol with 15 c.c. of 90 to 95 per cent. alcohol, 15 c.c. of water, and 10 c.c. of phosphoric acid syrup.

In examining a flour, 1 or 2 c.c. of this reagent are placed in a porcelain capsule, a pinch of the sample introduced, and the liquid warmed very slightly. After a few seconds any particles of sawdust present assume a rose tint which gradually darkens to carmine, whilst the cellulosic substances of the flour only become coloured subsequently, and usually very slightly.

The coloration given by maize and potato flour is much less pronounced than that of wood flour.

Attempts to make this reaction quantitative have only given uncertain results.

C. A. M.

Wines containing Citric Acid. R. Kunz. (*Zeit. für Untersuch. der Nahr. und Genussmittel*, ii., 692.)—Samples of so-called "wine composition" or "wine essence" examined at the Government Laboratory at Vienna were found to contain, besides tartaric acid, large quantities of citric acid. It is believed that these preparations are used not only in the production of artificial wines, but also for adulterating natural wines, and this opinion is strengthened by the fact that many wines, particularly those of Italian origin, have been found to contain considerable quantities of citric acid. The detection of citric acid in wines having thus become a matter of importance, the following method is proposed as being reliable and free from possible objections: 50 c.c. of the wine are mixed with 5 c.c. of a 20 per cent. solution of lead acetate. The precipitate is filtered off, washed, stirred up with 50 c.c. of water, and 3 c.c. of a 20 per cent. solution of sodium sulphide added to separate the lead. Should any turbidity be produced, a few drops of acetic acid may be added. The lead sulphide is then removed by filtration, the filtrate evaporated to dryness, the residue washed with the smallest possible quantity of water into a beaker, ammonia added to slight alkaline reaction, double the volume of ammonium chloride solution added, and the tartaric acid thrown down by calcium chloride. The large excess of ammonium chloride present hinders the precipitation of the citric acid. On now evaporating the filtrate from the calcium tartrate precipitate, the citric acid, if present, separates out in the form of crystals of calcium citrate. Or the filtrate from the calcium tartrate can be mixed with alcohol, the precipitate produced washed with alcohol, dissolved in a little hydrochloric acid, the solution made alkaline with ammonia and heated to boiling. This procedure has the advantage over the former that the removal of the ammonium chloride facilitates the precipitation of the calcium citrate. In either case the crystals of calcium citrate are identified by means of the microscope. H. H. B. S.

Estimation of Succinic Acid in Fermented Liquids. J. Laborde and L. Moreau. (*Ann. de l'Inst. Pasteur*, 1899, xiii., 657; through *Chem. Zeit. Rep.*, 1899, 283.)—Girard's process, though accurate, is tedious owing to the low temperature and slow speed of evaporation. If the temperature be raised, losses ensue, not because the succinic acid volatilizes, but because some of it combines with glycerin and does not take part in the final titration. The substance is preferably evaporated to dryness on the water-bath in presence of sand or fine shot, and the solid matter extracted with ether. The solvent is driven off, and in the dry extract free acid is estimated (1) by titration, (2) by saponification with an excess of potassium hydroxide. By adding the two yields together, the proportion of succinic acid is found; but the results are generally 0·1 to 0·2 gramme too high per litre of sound wine. Proper correction, however, can be made by determining the volatile acids. Free tartaric acid may pass into the ether; it must be converted into tartrate, estimated and deducted. If the original liquid contains more than 1 per cent. of sugar, extraction of the dry residue with ether is not complete; the sample must be evaporated only to a syrup, mixed with alcohol in amount corresponding with the

proportion of sugar, and then precipitated by the gradual addition of ether, repeating the whole process several times. F. H. L.

The Limits to the Detection of Malt Substitutes in Beer. E. Prior. (*Zeit. für Untersuch. der Nahr. und Genussmittel*, ii., 697-701.)—Experiments were made with the following mixtures: Forty-five grammes of malt and 5 grammes of potato starch, 45 grammes of malt and 5 grammes of rice, 40 grammes of malt and 10 grammes of potato starch, and 40 grammes of malt and 10 grammes of rice. Each mixture was mashed in the manner customary in making malt analyses, the mash filtered, the grain washed with hot water, and the wort boiled with 0·5 gramme of hops. After cooling and filtering, the proper quantity of yeast was added, and the liquid fermented. At the expiration of four days the beer was filtered and analysed. In order to saccharify the added starch and rice as completely as possible, both were boiled to paste, and the rice in addition was steamed for three hours at 3 atmospheres' pressure. The results are given in the following tables:

Analyses of the Potato Starch and Rice.

	In the Natural State. Potato Starch. Per cent.	In the Natural State. Rice. Per cent.		In the Dry State. Potato Starch. Per cent.	In the Dry State. Rice. Per cent.
Water	18·63	12·74	Water	—	—
Starch	77·30	74·00	Starch	95·00	84·80
Nitrogenous matter	—	8·25	Nitrogenous matter	—	9·91

Analyses of the Malts.

	Malt.	Malt + 10% Starch.	Malt + 20% Starch.	Malt + 10% Rice.	Malt + 20% Rice.
Time of saccharification ...	20-25 min.	45-50 min.	60 min.	40 min.	40-45 min.
Condition of the wort ...	clear	cloudy	cloudy	clear	clear
Maltose in the extract ...	67·11%	67·43%	69·03%	67·15%	68·30%
Nitrogen in the extract ...	0·645%	0·547%	0·279%	0·630%	0·650%

Analyses of the Beers.

	Malt.	Malt + 10% Starch.	Malt + 20% Starch.	Malt + 10% Rice.	Malt + 20% Rice.
Maltose in the extract ...	13·91%	17·29%	25·61%	15·20%	14·90%
Nitrogen in the extract ...	1·032%	0·955%	0·940%	1·33%	1·35%
Degree of fermentation ...	66·34%	65·30%	63·25%	66·98%	67·18%

The following conclusions were arrived at:

The time occupied in saccharifying is increased by the addition of starch or rice. With 20 per cent. of starch erythro-dextrin was still found at the expiration of an hour.

The percentage of maltose and achroo-dextrin in the unfermented extract is not

increased by the substitution of 10 per cent. of starch or rice, but is somewhat increased by a 20 per cent. substitution.

The percentage of nitrogen in the unfermented wort extract is perceptibly reduced by the substitution of 10 per cent. of starch, and is reduced by more than half by the substitution of 20 per cent. Ten or 20 per cent. of rice does not reduce the proportion of nitrogen in the unfermented wort extract.

The degree of fermentation of the beer is somewhat reduced by the substitution of 10 per cent. of starch, and more so by the substitution of 20 per cent. On the other hand, rice occasions a slight increase.

The percentage of maltose and achroo-dextrin in the beer extract is increased by starch or rice, especially by the former.

The nitrogen of the beer extract is reduced but little below 1 per cent., and certainly not below 0·9 per cent., by the substitution of even as much as 20 per cent. of starch. Rice increases the proportion of nitrogen in the beer extract.

H. H. B. S.

The Distinction of True Extract of Vanilla from Liquid Preparations of Vanillin. **W. H. Hess.** (*Journ. Amer. Chem. Soc.*, vol. xxi. [9], pp. 719-723.)

1. *Dilution Test.*—A small quantity of the tincture, agitated with two volumes of water, should furnish a flocculent reddish-brown precipitate, but only slight turbidity, since milkiness indicates foreign resin, as does also more than a slight turbidity ensuing on the addition of hydrochloric acid drop by drop to the diluted sample. If alkali has been used in preparing the extract, the turbidity is greater than otherwise, and the colour fades on addition of acid.

2. *Testing the Resin.*—Twenty-five c.c. of the extract under examination are evaporated on the water-bath to eliminate alcohol, and then made up to the original volume with water. The true resin forms an amorphous, flocculent, red to brown residue, separating out entirely on acidifying the solution with a little hydrochloric acid. It is soluble in caustic potash, giving a deep red solution, and is reprecipitated by acid. Unlike most other resins, this one gives little or no colour reaction with ferric chloride or hydrochloric acid in alcoholic solution.

3. *Colouring Matter.*—(*a*) Caramel: A portion of the filtrate from the resin is concentrated on the water-bath until about as dark as the original tincture. Caramel, when present, will be thrown down as a flocculent, yellowish-red precipitate on adding a few drops of strong hydrochloric acid and applying gentle heat. The precipitate is insoluble in water, strong alcohol, or ether, but soluble in dilute caustic potash, glacial acetic acid, or dilute alcohol. (*b*) Azo dyes: A portion of the filtrate is treated with ammonia—which deepens the colour of the true extract—and digested with zinc dust. If natural, the colour will revert to its original tint, or nearly so; but azo dyes will be decolorized, the colour returning on exposure to air, or on addition of hydrogen peroxide.

A portion of the original solution from the resin is tested for tannin, which should be present in small quantity only.

Organic acids and extractives are contained in considerable quantity in the pure

extract, and their precipitation by lead acetate forms a decisive test of the genuineness of the sample.

Coumarin, due to adulteration with Tonka extract, is detected by the method given in *Journ. Amer. Chem. Soc.*, vol. xxi., p. 256. C. S.

Estimation of Essential Oil in Cinnamon-Water. M. Duyk. (*Ann. de Chim. anal.*, 1899, iv., 223, 224.)—The method employed consists in treating the preparation with phenylhydrazine and weighing the insoluble cinnamyl hydrazone formed.

The cinnamon-oil of Ceylon and China consists almost entirely of cinnamic aldehyde, the proportion being as high as 90 per cent. in the Ceylon product.

The phenylhydrazine solution is prepared by dissolving 1 gramme of phenylhydrazine hydrochloride and 1·5 gramme of potassium acetate in 10 c.c. of water.

A sufficient quantity of this reagent is added to the cinnamon-water to cause complete precipitation of the cinnamic aldehyde at the ordinary temperature, and the precipitate collected on a filter, washed, dried on a porous plate under a clock-glass, and weighed.

In a test experiment the author dissolved 0·1 gramme of cinnamic aldehyde in 100 c.c. of water, and obtained 0·162 gramme of the hydrazone as against the theoretical amount 0·167 gramme. In an officinal sample the weight of the precipitate was 0·175 gramme, corresponding with rather more than 0·10 gramme of cinnamic aldehyde.

The distilled waters of certain other essential oils, such as peppermint, rose, orange flowers, and camomile, do not yield any precipitate with phenylhydrazine, but the method is applicable to the estimation of benzaldehyde in cherry-laurel-water.

As a general rule the presence of small amounts of alcohol in the waters do not interfere with the determination. C. A. M.

Estimation of Santonin. J. Katz. (*Arch. Pharm.*, 1899, ccxxxvii., 245; through *Chem. Zeit. Rep.*, 1899, 233.)—The author rejects the processes hitherto employed, and proposes the following: Ten grammes of coarsely powdered santonica are extracted with ether in a Soxhlet for two hours. The solvent is distilled off, and the residue is cohobated for fifteen or thirty minutes with a solution of 5 grammes of crystallized barium hydroxide in 100 c.c. of water. After cooling, the liquid is saturated with carbon dioxide till it has an acid reaction, filtered quickly, and the carbonate washed twice with 20 c.c. of water. The filtrate is concentrated on the water-bath to about 20 c.c., mixed with 10 c.c. of 12·5 per cent. hydrochloric acid, and heated for another two minutes. The liquid is brought into a separating funnel, the crystals in the basin being dissolved in 20 c.c. of chloroform; and basin, funnel, and filter are washed twice with 20 c.c. of the same solvent. The liquid is evaporated, and the residue boiled under a vertical condenser for ten minutes with 50 c.c. of 15 per cent. alcohol; the solution is filtered into a tared flask, and the insoluble matter treated twice again with 10 c.c. of boiling 15 per cent. spirit. After standing twenty-four hours, the flask and its contents are weighed, the alcohol is filtered

through a weighed paper, flask and paper being rinsed with 10 c.c. of the same spirit (which is not included in the subsequent calculation). The filter is finally dried and weighed; and to the weight of santonin so obtained a correction of 0·006 gramme is added for every 100 grammes of filtrate. F. H. L.

The Assay of Cacodylates. H. Imbert and A. Astruc. (*Journ. Pharm. Chim.*, 1899, x., 392-395.)—The fact that cacodylic acid—$AsO\begin{matrix}OH\\CH_3\\CH_3\end{matrix}$—behaves as a neutral body with the indicator helianthine A, but is monobasic with phenol-phthalein, affords a rapid method of estimating it.

Thus, in the case of sodium cacodylate, which is the salt most frequently met with in commerce, 1 molecule of acid should be required to render the salt neutral to helianthine. But in practice the authors have found that the samples they have met with have not the theoretical neutrality to phenol-phthalein, but are, on the contrary, markedly acid. This they consider is probably due to the dissociation of the salt by water in a manner analogous to that of bimetallic glycerophosphates.

In making an assay 1·6 gramme of sodium cacodylate is dissolved in 100 c.c. of water, giving a solution containing one-tenth of the molecular weight per litre. Ten c.c. of this liquid are neutralized with standard alkali, with phenol-phthalein as indicator, and then titrated with decinormal acid with helianthine as indicator. The number of c.c. required multiplied by 10 gives the percentage of pure sodium cacodylate in the salt.

In different commercial samples the authors found by this method from 75·20 to 92·80 per cent. of pure sodium cacodylate, and from 9·66 to 27·60 per cent. of free cacodylic acid.

Cacodylic acid and cacodylates do not give the characteristic reactions of arsenates. C. A. M.

ORGANIC ANALYSIS.

The Detection of Acetylene by Means of an Ammoniacal Solution of Copper and Hydroxylamine. L. Ilosvay and N. Ilosvay. (*Berichte*, 1899, xxxiii., 2697-2699.)—The colourless solution obtained by reducing an ammoniacal solution of cupric sulphate with hydroxylamine can be used as a reagent for acetylene, and the author has made experiments to determine under what conditions it is most sensitive and yields a copper acetylide of constant colour.

He finds that the sensitiveness of the liquid is dependent on the amount of ammonia and hydroxylamine it contains. If the proportion of ammonia be only just sufficient to dissolve the copper salt and the hydroxylamine only sufficient to just decolorize the liquid, the precipitate formed by acetylene is at first yellowish-brown and subsequently green. If, on the other hand, the ammonia be in considerable excess, and a correspondingly large amount of hydroxylamine present, the colour of the precipitate varies between a light and dark onion red. If there be too

little ammonia, a light yellow pulverulent substance (probably copper suboxide or cuprous hydroxide) is formed on the surface. This, however, does not destroy the sensitiveness of the reagent.

As a general rule, the solution should contain from 2·5 to three times the amount of ammonia absolutely necessary, and from fifteen to eighteen times the quantity of hydroxylamine required to reduce the copper compound.

The author gives the following directions for preparing the reagent (50 c.c.) from the chloride, nitrate, or sulphate of copper:

1. Cupric chloride ($CuCl_2\,3H_2O$), 0·75 gramme; ammonium chloride, 1·5 gramme; ammonium hydroxide (20 to 21 per cent. NH_3), 3 c.c.; and hydroxylamine hydrochloride, 2·5 grammes.
2. Cupric nitrate ($Cu(NO_3)_2.5H_2O$), 1 gramme; ammonium hydroxide, 4 c.c.; and hydroxylamine hydrochloride, 3 grammes.
3. Crystalline cupric sulphate, 1 gramme; ammonium hydroxide, 4 c.c.; and hydroxylamine hydrochloride, 3 grammes.

In each case the copper salt is dissolved in a little water, the ammonium hydroxide added, then the hydroxylamine hydrochloride, and the solution shaken and made up to 50 c.c.

The solutions remain stable for about three days, but on the fourth day become less sensitive, and subsequently begin to deposit a light red precipitate, whilst after fourteen days the metal is rapidly reconverted into the cupric state.

The presence of acetylene in coal-gas can be readily demonstrated by means of these solutions. A few c.c. of the reagent are placed in a 500 c.c. stoppered cylinder, the gas passed in until the liquid becomes rose-coloured, and the cylinder then closed and shaken, an immediate red precipitate being formed. C. A. M.

Estimation of Invert Sugar in Presence of Cane-Sugar. H. Jessen-Hansen. (*Meddelelser fra Carlsberg Lab.*, 1899, iv. [4], 314; through *Résumé in French, ibid.*, p. 193.)—Bruhns has recently objected (ANALYST, 1898, xxiii., 297) to Kjeldahl's method of estimating invert and other sugars with alkaline copper solutions (ANALYST, 1895, xx., 227), on the ground that it is incorrect when cane-sugar is simultaneously present, for the latter precipitates a large and variable amount of copper, which vitiates the results of the analysis. Jessen-Hansen remarks that Kjeldahl was not dealing in his original communication with such materials as may contain cane-sugar, therefore the criticism is somewhat unjust; but in order to render Kjeldahl's process available when reducing sugars are mixed with large quantities of cane-sugar, he has reinvestigated the whole question. He finds that when 30 c.c. of Fehling's solution, prepared according to Kjeldahl's directions, but containing double the usual amount (10·4 instead of 5·2 grammes) of Rochelle salt are used, and when the time of boiling in a current of hydrogen is limited to five minutes, the weight of copper thrown down by 10 grammes of cane-sugar is lowered from between 102 and 114 milligrammes to about 14 milligrammes, so that if the period of ebullition is governed exactly by means of a watch having a second-hand, concordant results can always be obtained. It follows, therefore, that the figures given by Kjeldahl (ANALYST, 1895, *ubi sup.*) for

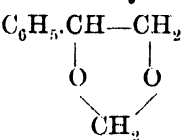
invert sugar require modification in presence of cane-sugar; and the present author accordingly appends to his paper a lengthy table for converting the weight of copper deposited into invert sugar (*a*) when the latter exists alone, and (*b*) when it is mixed with different proportions of cane-sugar. The figures for pure invert sugar also are not quite the same as those already recorded. F. H. L.

The Odoriferous Principle of Jasmine Flowers. A. Verley. (*Bull. Soc. Chim.*, 1899, xxi., 226-232.)—Numerous attempts have been made to isolate the essential oil of jasmine flowers by distillation with steam, but these have been unsuccessful owing to the quantity of oil being very small, and to the fact that it is completely decomposed on boiling with acidulated water.

The author has obtained it by *enfleurage* in the cold. The fat saturated with the perfume was beaten with vaseline-oil, and the latter extracted with acetone. On evaporating the acetone under reduced pressure a clear reddish-brown oil was left.

A fractional distillation of 35 grammes of this substance under reduced pressure yielded 19 grammes between 70° and 180° C. under 15 mm. pressure, and a residue of 14 grammes of resinous matters. These 19 grammes on re-fractionation under 12 mm. pressure gave the following fractions: 70° to 100° C., 2 grammes; 100° to 101° C., 11 grammes; 101° to 110° C., 3 grammes; and 110° to 180° C., 2 grammes. The fraction distilling at 100° to 101° C. was a very mobile, slightly yellow liquid, with an intense odour of jasmine. It had a density of 1·1292 at 0° C., and a composition corresponding with the formula $C_9H_{10}O_2$.

To this substance, which was not quite pure, the author gave the name "jasmal. From its reactions it appeared to be the methylene acetal of phenylglycol.



This conclusion was confirmed by the synthetical preparation of that acetal. Fifty grammes of phenylglycol, 300 grammes of water, 125 grammes of sulphuric acid, and 100 grammes of formaldehyde were heated together on the water-bath, and the resulting clear supernatant oil removed by ether and rectified under reduced pressure. It boiled at 100° C. under 12 mm. pressure, and at 218° under the ordinary pressure. Its density was 1·334 at 0° C. In the pure state it had the characteristic odour of jasmine, and all the properties of the natural jasmal. When boiled with acidulated water phenylglycol and formaldehyde were regenerated.

Homologous compounds were obtained in the same manner, another aldehyde of the fatty acid series being substituted for formaldehyde, but these had not so pronounced an odour of jasmine. C. A. M.

Essential Oil of Jasmine Flowers. A. Hesse and F. Müller. (*Berichte*, 1899, xxxii., 565-574 and 765-779.)—The authors state that for some time past they have extracted the essential oil of jasmine by a method analogous to that described by

Verley (see preceding abstract), but have found that it is preferable to purify the oil by distillation in steam rather than under reduced pressure.

In this way they have determined the amount and characteristics of the oil recovered from numerous samples of jasmine pomade from the harvests of 1896 and 1897, and give the results of ten representative cases.

In these the amount of ethereal oil obtained from 1 kilo of pomade varied from 3·95 to 5·95 grammes, and had the following characteristics:

Specific gravity at 15° C., 1006—1018; optical rotation, +2° 30′ to +3° 30′; ester content in terms of linalyl acetate, 90·3—95·4 per cent.; in terms of benzyl acetate, 69·1—73·0 per cent.

One of the authors showed in a paper some years ago that the high percentage of esters in ethereal jasmine-oil was to be attributed to the presence of large quantities of benzyl acetate.

Since Verley has stated that the odoriferous principle in jasmine-oil is phenylglycol-methylene acetal, the authors have examined products obtained in exactly the same manner as described by Verley. Phenylglycol-methylene acetal prepared by the condensation of phenylglycol and formaldehyde, by means of dilute sulphuric acid, is not acted upon by alkalies; but on treatment with oxalic acid it is decomposed, with the liberation of phenyl glycol, which can readily be identified by its characteristic crystalline form, and the formation of phenyl acetaldehyde on heating it with dilute sulphuric acid.

Having proved that in this way it was possible to detect small quantities of the acetal in the presence of linalol and of benzyl acetate (in large excess), the authors tested the different fractions yielded by the distillation of 60 grammes of jasmine-oil under reduced pressure, but in none of them could any trace of phenylglycol-methylene acetal be detected.

The odoriferous principle was found to be due in the main to two substances, which together only amounted to 5 per cent. of the total volatile oil, and neither of which was Verley's acetal.

In order to determine the amount of the various constituents in jasmine-oil, a method based on the behaviour of these substances towards permanganate was devised.

It was proved experimentally that linalol, when thus oxidized, yielded carbonic and acetic acids, but no solid acid. Benzyl alcohol, however, was converted into benzoic acid, and in a test experiment 94·5 per cent. of the theoretical quantity was obtained. Crude linalyl acetate, prepared by boiling linalol with acetic anhydride and sodium acetate, behaved in an analogous manner to free linalol, and yielded carbonic and acetic acid and a small amount of an oily, soluble acid, but no solid acid. Benzyl acetate, in the absence of alkali, was not attacked by permanganate.

As a test of the applicability of this method to the examination of jasmine-oil, a mixture was prepared consisting of 30 per cent. of benzyl alcohol, 50 per cent. of benzyl acetate, 10 per cent. of linalol, and 10 per cent. of crude linalyl acetate (containing about 59 per cent. of that acetate). This mixture had a saponification value of 201, the calculated value being 200·2.

The mixture during oxidation was kept as neutral as possible by the addition of dilute sulphuric acid, and the temperature was maintained at from 15° to 20° C. by

adding ice. The permanganate was added at first in a 3 per cent. solution, and subsequently in the form of a fine powder, which was introduced little by little, with constant agitation, until the colour remained permanent for about an hour.

The excess of permanganate was removed with bisulphite, and the oxidized liquid extracted three or four times with ether. The acid oxidation-products were removed from the ethereal extract by shaking the latter with a dilute solution of sodium hydroxide. The residual ethereal layer was washed with water, dried with calcium chloride, and evaporated to dryness at 40° C. in a weighed and exhausted flask. The residue was benzyl acetate.

The alkaline extract was added to the original residue from the ethereal extraction, the mixture rendered slightly alkaline, filtered, the filtrate concentrated by evaporation and freed as far as possible from inorganic salts by the addition of alcohol, and the benzoic acid determined by rendering the liquid acid and filtering.

In this way, from 20 grammes of the above mixture, 9·8 grammes of benzyl acetate (= 98 per cent. of the theoretical amount) and 6·6 grammes of benzoic acid (97·5 per cent. of the theoretical quantity) were obtained. From these results the authors concluded that the method was capable of determining the amount of these substances within 2 to 4 per cent. of the real quantity.

Having proved qualitatively that the different fractions obtained in the fractional distillation of essential oil of jasmine consisted chiefly of linalol, linalyl acetate, benzyl acetate, and benzyl alcohol, the authors employed the oxidation method described above for a quantitative estimation, in which the percentage of linalyl acetate was calculated from the difference between the ester content of each fraction, and the amount of benzyl acetate actually found.

From the results obtained in the examination of the fractions and of the original crude substance, they came to the conclusion that the essential oil of jasmine flower contains in round numbers:

Benzyl acetate, 65; linalyl acetate (and other terpene alcohol esters), 7·5; benzyl alcohol, 6; other odoriferous constituents, 5·5; and linalol (and other substances), 16 per cent.

Hesse (*Berichte*, 1899, xxxii., 2611-2620) has continued the research on this subject commenced by himself and Müller. He finds that, in addition to the oxygen compounds described in the former papers, certain nitrogenous compounds are present and have a considerable influence on the nature of the perfume.

Of these the more important is indol ($C_8H_{17}N$), which can be isolated by precipitation with picric acid. It amounts to about 2·5 per cent. of the crude oil.

The author also states that the constituent of the oil which reacts with picric acid and the "aldehyde" of jasmine-flower-oil isolated by means of sodium bisulphite are identical.

The methyl ester of anthranilic acid ($NH_2.C_6H_4.CO_2.CH_3$) is also present in small amount (not exceeding 0·5 per cent.). This ester, which has also been found by Walbaum and Erdmann in neroli-oil, gives in dilute solution the blue fluorescence of jasmine-flower-oil. When present in greater quantity than 1 per cent., it can be precipitated as a sulphate by adding a mixture of sulphuric acid and ether. Its influence on the jasmine perfume is much less than that of the indol.

A third new constituent has been isolated from the fraction of the jasmine-oil distilling between 105° and 107° under 5 mm. pressure. This is a ketone of the formula $C_{11}H_{16}O$, to which the author has given the name of *jasmone*. It is a light yellow oil, becoming darker on keeping, and having an intense odour of jasmine. It has a specific gravity of 0·945 at 15° C., and boils at 257° to 258° C., under a pressure of 755 mm. It can readily be isolated as a semi-carbazone, which when purified by one crystallization from boiling alcohol melts at 204° to 206° C. The pure jasmone oxime ($C_{11}H_{16}NOH$) crystallizes from dilute alcohol in fine needles which melt at 45° C., and are volatile in a current of steam.

From the results of the investigations described in this and in the former papers, the author gives the following as the composition of the ethereal oil of jasmine flowers:

Jasmone, 3·0; indol, 2·5; methyl ester of anthranilic acid, 0·5; benzyl acetate, 65·0; linalyl acetate, 7·5; benzyl alcohol, 6·0; and linalol, 15·5 per cent.

C. A. M.

Bishop's Reaction for Sesamé Oil. H. Kreis. (*Chem. Zeit.*, 1899, xxiii., 802.)—In 1892 Ambühl mentioned that on testing an old rancid sample of sesamé with the Baudoin reaction, he obtained a blue colour instead of the usual red, a peculiarity which has not hitherto been explained. Quite recently the present author, examining a saccharine secretion from larch needles with sesamé and hydrochloric acid in order to test for sugar, obtained a blue-violet colour when the oil was rancid, but the normal red tint when a fresh specimen of sesamé was substituted. It seemed, therefore, that this blue might be a mixture of the Baudoin red with the green, which, as Bishop discovered (*Journ. Pharm. Chim.*, 1889, [5], xx., 244), is produced when old sesamé oil is shaken with 1·19 hydrochloric acid; and experiments showed that, by mixing together the coloured acids from the two tests, Ambühl's blue could be imitated in its various shades. The blue colour with rancid sesamé appears more distinctly when sugar is used than with furfural. Bishop's test gives at first a yellowish-green tint, which gradually changes to bluish-green, while the oil finally becomes pale violet. In the presence of resorcinol or phloroglucinol, Bishop's acid turns violet or orange-red, but other phenols exert no influence on the colour.

Bishop recommended his test as a means of judging the age of sesamé oil, but as the reaction seems to be caused by oxidized products, substances similar to which may be present in other oils, it is not surprising that an analogous green colour is produced when certain other rancid fats mixed with fresh sesamé are agitated with strong hydrochloric acid. For instance, an old olive oil (acid No. 5) gave no colour on shaking with acid, but after being mixed with an equal volume of a sesamé oil, which itself yielded no reaction, the whole immediately produced a green tint. The same phenomena were observed with old butter (acid No. 6) and lard (acid No. 2); neither coloured the acid alone, but with indifferent sesamé oil both gave the Bishop reaction. The investigation of this subject is being continued; in the meantime, it seems that Bishop's reaction is shown most strongly by those oils which still possess a normal acid number, but which are shown to be rancid by their taste. F. H. L.

Some New Reactions for Sesamé Oil. F. Breinl. (*Chem. Zeit.*, 1899, xxiii., 647.)—The furfural test for sesamé (ANALYST, 1896, xxi., 135) appears not only in presence of strong hydrochloric acid, but also if other dehydrating agents are used, such as sulphuric acid diluted with water, or preferably with glacial acetic acid. Other aldehydes—presumably all the aromatic compounds—give similar reactions; but the exact colours vary with the reagent employed. Benzaldehyde and cuminol yield an orange; *o*-nitrobenzaldehyde, an orange-yellow; *p*-nitrobenzaldehyde, a yellow; cinnamic aldehyde, *o*- and *p*-hydroxybenzaldehyde, vanillin, and piperonal all give a violet-red tint. Neither formaldehyde nor acetaldehyde produces a colour with sesamé oil; but as cinnamic aldehyde does, it would seem that unsaturated fatty aldehydes should behave analogously. Crotonic aldehyde, even in very dilute solution, yields an orange coloration.

As these colours are very similar to those given by furfural, etc., with albuminous substances, it would appear probable that the actual cause of the reaction might be a nitrogenous albumin-like body in the sesamé oil. Evidence of this cannot be obtained; nevertheless, repeated extraction of sesamé with strong hydrochloric acid removes from the oil the power of yielding colours, while the aqueous liquid shows a noteworthy colour. The acid extracts also contain a minute trace of nitrogen. Inasmuch as animal fats have been shown to contain albuminoids, it is not improbable that vegetable fats, especially those which have not been refined with strong acids, should contain proteids; and as albuminoids give the above-mentioned furfural reaction, it is not surprising that some samples of olive and castor oils have responded to the Baudoin test. A few specimens of the latter have also reacted with piperonal and vanillin; but the colours were so faint as to be overshadowed by the addition of 1 per cent. of sesamé.

Experiments have been made to determine the comparative delicacy and general utility of the several aldehydes as reagents for sesame oil; and seeing that many fats give red or brownish colours on shaking with strong hydrochloric acid alone, those substances are preferable which yield tints bluer than that produced by furfural. On the whole, *p*-hydroxybenzaldehyde, vanillin, and piperonal are to be recommended; and their alcoholic solutions have the advantage of being permanent for months when exposed to light. On account of their higher molecular weights, however, they should be used as 3 per cent. solutions. F. H. L.

Two New Colour Reactions for Sesamé Oil. J. Bellier. (*Ann. de Chim. anal.*, 1899, iv., 217-220.)—On shaking equal parts of this oil and a reagent consisting of sulphuric acid, 100 c.c.; water, 50 c.c.; and formaldehyde (40 per cent.), about 10 c.c., an emulsion is obtained which gradually assumes an intense and stable bluish-black colour; whilst with olive, cottonseed, earthnut, poppy, and nut oils the emulsion formed is of a more or less pronounced yellow. By this means it is possible to detect as little as 1 per cent. of sesamé oil in olive oil even in the case of Tunis oils, which give a relatively dark coloration with hydrochloric acid and furfural, but behave like other pure olive oils with this reagent.

The following is said to be an equally characteristic test: Two c.c. of the oil are

shaken with 2 c.c. of crystallizable benzene saturated with resorcinol, and 2 c.c. of nitric acid free from nitrous vapours (specific gravity 1·38).

With sesamé oil the mixture assumes an intense bluish-violet colour almost immediately, and the acid which separates has an intense bluish-green colour which lasts for five or ten minutes.

In the case of olive oil the mixture sometimes remains colourless, but generally, and especially with Tunis oils, has a more or less pronounced violet tint, while the acid which separates is at first pale yellow and finally orange yellow.

Earthnut, cottonseed, poppy, nut, and linseed oils yield an intense violet-blue mixture, but, unlike sesamé oil, do not give a green-coloured acid on the layers separating. With olive oil containing 1 per cent. of sesamé oil the green colour of the acid layer is still marked after several minutes. C. A. M.

INORGANIC ANALYSIS.

Solubility of Lead Sulphate in Ammonium Acetate. J. C. Long. (*Amer. Chem. Journ.*, vol. xxii. [3], pp. 217, 218.)—A series of experiments made show that the maximum solvent effect is exerted on lead sulphate when the ammonium acetate solution is of 31 to 37 "volume per cent." (29 to 34·3 per cent.) strength; therefore, to dissolve this lead salt, it should be boiled in 10 c.c. (for 1 gramme) of a 33 per cent. solution of the acetate (specific gravity 1·07), and filtered promptly. It is found that, in recovering the lead sulphate, the evaporation and expulsion of the ammonium acetate without loss is greatly facilitated by an addition of 0·5 gramme of sulphuric acid. C. S.

Analysis of White Metal. H. Nissenson. (*Chem. Zeit.*, 1899, xxiii., 868.)—Under the name of "metallic packing" for engine glands, etc., two classes of alloys are frequently to be met with, containing about 80 per cent. of lead with (*a*) 6 per cent. of antimony and 12 per cent. of tin, or (*b*) with 12 to 15 per cent. of antimony and 3 to 5 per cent. of tin. To analyse them, 1 gramme of the sample is mixed with 2 to 4 grammes (4 grammes for class *a*, 2 grammes for class *b*) of tartaric acid, 12 c.c. of water, and 4 c.c. of 1·4 nitric acid, warming till solution is effected. Four c.c. of strong sulphuric acid are then added to precipitate the lead, evaporating in such fashion that the nitric acid is driven off but the tartaric acid not decomposed. To attain this object, the liquid is heated in an Erlenmeyer flask as long as the red vapours are produced; and in order to watch the process conveniently, the nitrous acid is blown out of the vessel with a bent piece of tubing periodically. Immediately the fresh bubbles of gas are white, the flame is removed; a little water is introduced, and the lead sulphate is collected. The filtrate is made alkaline with sodium hydroxide, 50 c.c. of a cold saturated solution of sodium sulphide are run in (any copper thrown down being removed), and the whole is electrolysed at 80° C. with a current of 1·5 ampères at 3 volts. The liquid is poured off from the antimony, which is rinsed in a little water and dried. The sodium sulphide in the solution is next converted into ammonium sulphide by treatment with 25 grammes of ammonium

sulphate, and the tin is deposited with the same current, and at the same temperature as before. F. H. L.

Determination of Chromium in Steel. R. W. Mahon. (*Journ. Amer. Chem. Soc.*, vol. xxi. [11], pp. 1057-1060.)—A modification of McKenna's method gives good results, 3 grammes of the substance being dissolved in 50 c.c. of concentrated hydrochloric acid, and boiled down to a moist cake. This is boiled for a few minutes in 50 c.c. of concentrated nitric acid, and, after cooling in the dark, is treated with 4 grammes of potassium chlorate, and concentrated to 25-30 c.c. After dilution to 300 c.c. with water, 15 c.c. of ammonia, specific gravity 0·90, are stirred in, and the (cool) solution passed through a ribbed double filter-paper to remove oxides of manganese. The filtrate and (cold water) washings are made up to about 450 c.c., and titrated with standard ferrous ammonium sulphate and potassium permanganate, preferably standardized by standard potassium bichromate. If the removal of hydrochloric acid has been completely effected by nitric acid, about 2 grammes of potassium chlorate will suffice to oxidize the chromium present; but this salt is a more convenient agent than nitric acid for eliminating the final traces of hydrochloric acid. The solution must be dilute and cold, as well as free from too great an excess of nitric acid, in order that reduction of the chromic acid by the filter-paper may be prevented.

The manganese oxides on the filter may be utilized for the gravimetric determination of this metal, the precipitate being dissolved in hydrochloric acid, the iron thrown down as basic acetate, and the manganese as manganous ammonium phosphate.

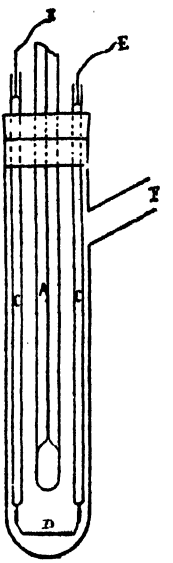
C. S.

Estimation of Chromium in Iron and Steel. E. Döhler. (*Chem. Zeit.*, 1899, xxiii., 868.)—When a hydrochloric acid solution of iron is precipitated with barium carbonate, not all the chromium is thrown down the first time; if the sample contains much chromium (4 per cent.), even a third treatment is necessary to recover it all. 2·5 to 5 grammes of the iron are dissolved by heating with 30 c.c. of 1·19 hydrochloric acid and 100 c.c. of water in a 750 c.c. flask closed with a valve-stopper. The liquid is diluted to about 600 c.c. with cold water, and a cream of barium carbonate is added until, after half an hour's standing, an excess is still noticeable. The precipitate, which should be slightly yellow owing to traces of iron, is allowed to rest in the well-closed flask for twelve hours, then filtered off through a covered funnel, and the filtrate is treated again with barium carbonate precisely as before. The mixed precipitates are ignited in porcelain with alkali carbonates and potassium nitrate, at first over a Bunsen flame, and finally for half an hour before the blowpipe. The mass is extracted with hot water, filtered from iron and barium, freed from silica by evaporation with hydrochloric acid, again filtered, and the chromium and aluminium separated as usual. Neither silver, nickel, nor platinum crucibles must be used, and a fresh porcelain one should be employed each time. Too large an excess of barium carbonate is to be avoided, and the substance should be very finely subdivided.

F. H. L.

APPARATUS.

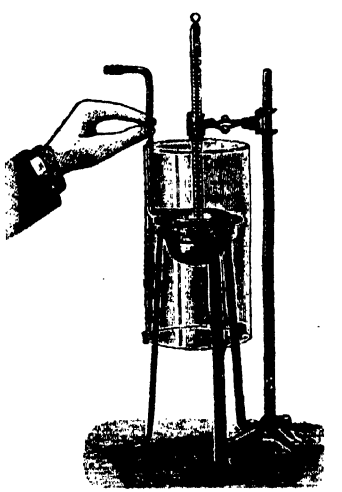
A Simplification of Beckmann's Boiling-point Apparatus. **S. L. Bigelow.** (*Amer. Chem. Journ.*, vol. xxii. [4], pp. 280-287.)—The substitution of electric heating for gas enables the sealed platinum wire, the glass beads, asbestos stove, outer jacket, and its attached reflux condenser, of the Beckmann apparatus to be dispensed with, and also obviates danger arising from breakage and fire in the case of inflammable solvents.



In the drawing, A represents the thermometer, and C,C two mercury tubes for conveying current to the platinum coil D, which is made of 0·1 millimetre wire wound to a diameter of about 1 millimetre, and measures 1½ to 2 centimetres in length, the free ends being preferably sealed into the mercury tubes. To replace the outer jacket employed by Beckmann, the vessel may be immersed in a battery jar well lined with felt; a Dewar vacuum jacket would, however, be preferable, to facilitate inspection.

A current of about 1·8 ampères from an incandescent lamp circuit will boil 30 c.c. of water in five minutes; and 3 cells of a storage-battery furnished the author with good results in the case of readily volatile solvents. Care is necessary to avoid any great perpendicular length of wire at the ends of the coil, or the wire may become enveloped in vapour and grow red-hot. C. S.

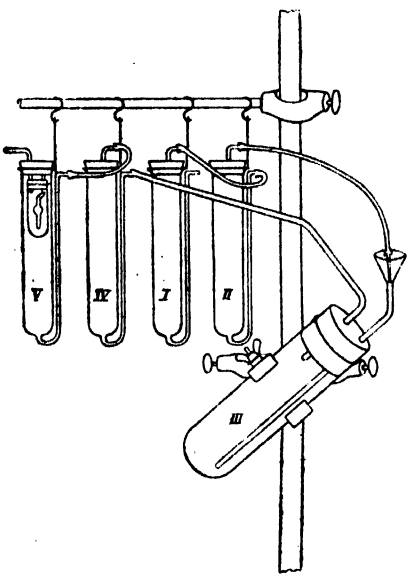
Apparatus for determining the Flash-point of Lubricating Oil. **R. Kissling.** (*Chem. Zeit.*, 1899, xxiii., 800.)—As shown in the illustration, this apparatus consists of a copper basin, 50 millimetres deep and 70 millimetres wide, filled with sea-sand, and containing a porcelain crucible to hold the oil, 45 millimetres deep and 40 millimetres in diameter. The crucible is sunk into the sand till it projects 10 millimetres, and it is charged with oil to the same level. The shade is made of heat-resisting glass, 250 millimetres high and 100 millimetres in diameter; it protects the apparatus from draughts, and prevents the igniting flame from being blown out by sudden gusts of air. The main burner is of such power that the oil rises about 2° in temperature in thirty seconds, and tests are made at the same intervals of time, commencing as soon as the sample reaches 20° or 30° below its expected flashing-point. A copper lid, provided with a slit for passing over the thermometer stem, and with a handle, is used to extinguish the flame of the burning oil.



F. H. L.

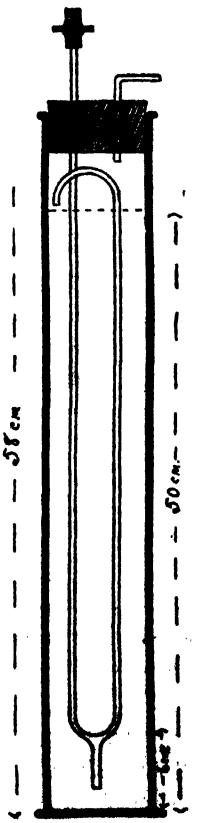
An Asbestos Filter-Tube. O. Lohse. (*Ber.*, 1899, xxxii., 2142.)—This consists of a piece of combustion tubing 12 centimetres long and 15 millimetres in diameter, expanded for about one-quarter of its length to a diameter of 20 millimetres, and closed like a test-tube at the end of its narrower portion with a hemispherical bottom, which is perforated with a number of small holes. It is charged with asbestos and fixed to a tube funnel for filtrations in the same manner as a Gooch crucible, while the shoulder enables it to be supported safely in a desiccator provided with a suitably perforated tray. The tube can be fitted with a glass stopper, if so desired, and by applying a cork and leading tube to its mouth the contained precipitate can be ignited in a current of gas. The author also describes various desiccators of somewhat obvious design, one of which holds caustic potash as well as calcium chloride. F. H. L.

An Improved "Potash-bulb," and a Simple Apparatus for estimating Carbon Dioxide by Absorption. R. Schaller. (*Zeits. angew. Chem.*, 1899, 878.)—The kind of vessel employed by the author to absorb carbon dioxide is shown by the tube marked V in the annexed illustration. He claims for it that useful space is raised to a maximum, that useless space is reduced to a minimum; that the tube has very little external surface, but a high absorbing power. The wide limb of the U is half filled with hollow round glass balls about 2 millimetres in diameter, and it contains about 10 c.c. of 50 per cent. potassium hydroxide. At the top, suspended by means of the tube through which the air passes out of the complete apparatus, hangs a small test-tube 8 centimetres long, filled with 1 c.c. of strong sulphuric acid; the tube whereby the air enters the latter is formed as shown, and extends to just below the level of the liquid. The carbon dioxide enters through the narrow limb of the U, and travels upwards through the moistened bulbs. The illustration as a whole represents apparatus for determining carbon dioxide by treating carbonates with acid in the vessel III. I and II, filled like V, free the current of air from carbonic acid; IV is a drying tube similarly filled with sulphuric acid. For exact work V is used in duplicate. The joint at the funnel is made with a tapering glass tube which enters the parallel funnel-tube; it is rendered air-tight by a water-seal. The current of air is aspirated rather than driven through the apparatus; and to ensure regularity of passage, either the final exit tube or both the exit and the first admission tubes are contracted to a fine point. The manipulation is obvious. F. H. L.



Platinum Gauze as a Cathode for Electrolytic Analysis. C. Winkler. (*Ber.*, 1899, xxxii., 2192.)—A piece of platinum gauze 10·5 centimetres long by 5·5 centimetres wide, composed of wires 0·12 millimetre in gauge, and with 250 meshes per square centimetre, has its edges bent over for a width of 2 or 3 millimetres so as to strengthen it, and is then coiled into an open (C-shaped) cylinder 5 centimetres high by 3·5 centimetres in diameter, leaving a slit 5 millimetres wide for the insertion of the anode. This form of cathode weighs only 13 grammes, and is capable of bearing its own weight of copper without any danger of the deposit falling off when washed. Much higher current-densities can be employed than formerly, reducing the time required to electrolyse a solution to about one-quarter of its usual length. Copper may be safely thrown down from its sulphate with a current of 0·5 ampère (density, $D_{100} = 5·1$ ampères), and 13 grammes can be deposited in sixteen or eighteen hours. Very gratifying results have also been obtained with silver, nickel, cobalt, zinc, and antimony. F. H. L.

A Dissolver. A. J. Hopkins. *Amer. Chem. Journ.*, vol. xxii. [5], pp. 407-410.)—An ordinary glass cylinder of suitable capacity may be used, that shown in the figure holding 2,200 c.c. A straight 6-millimetre tube is passed through one hole in the stopper, and connected below with a Y-tube, the other upper limb of which is connected to an upcast tube, bent over at the top above the surface of the dissolving liquid. When the cylinder is charged with solvent and the salt to be dissolved, air is drawn through the apparatus by an aspirator connected with the short bent tube on the right, and the inflowing current carries up with it from the Y-tube small columns of the saturating solution drawn through the lower limb, which are discharged at the top and are automatically replaced by fresh portions of the solvent descending the cylinder.



The apparatus is very rapid in action, 30 grammes of copper sulphate having been dissolved in 200 c.c. of water at 19° C. in forty minutes; and with a larger cylinder 1 kilo of ammonium carbonate was dissolved in 4 litres of water and 1 litre of ammonia in about half an hour. C. S.

INSTITUTE OF CHEMISTRY OF GREAT BRITAIN AND IRELAND.

THE following is a list of the names of the candidates who passed the examination of the Institute of Chemistry held in January, 1900:

INTERMEDIATE EXAMINATION.—Aston, F. W., Mason University College, Birmingham. Austin, J. H., Mason University College, Birmingham. Bailey, H. J., University College, Sheffield. Bayly, H. G., King's College, London. Clarke, G., jun., University College, Nottingham. Danks, M. W., Mason University College, Birmingham. Blake Smith, R. E., B.Sc. (Lond.), University College, London. Taylor, Thomas, Glasgow and West of Scotland Technical College. Webster, John, Mason University College, Birmingham. Woodman, W. E., King's College, London.

A.I.C. EXAMINATION (old regulations).—Brooke, J. R., Pharmaceutical Society's Laboratories and King's College, London. Priest, M., Finsbury Technical College. Tickle, T., Pharmaceutical Society's Laboratories, King's College and University College, London.

FINAL A.I.C. EXAMINATION.—*In Branch A (Mineral Chemistry)*: Fyffe, W. A., University College, Dundee. Wright, L. V., B.A., Sidney College, Cambridge. *In Branch B (Metallurgical Chemistry)*: Winch, H. J., A.C.G.I., City and Guilds of London Central Institution and Finsbury Technical College. *In Branch C (Physical Chemistry)*: Price, T. S., B.Sc. (Lond.), Ph.D. (Leipzig), Mason University College, Birmingham, and the Universities of Leipzig and Stockholm. *In Branch D (Organic Chemistry)*: Edmed, F. G., Assoc.R.C.Sc. (Lond.), B.Sc. (Lond.), Royal College of Science, London. Eynon, L., Finsbury Technical College. Hall, H., University College, Nottingham. Shedden, F., B.Sc. (Lond.), Mason University College, Birmingham. *In Branch E (the Analysis of Food and Drugs, including an Examination in Therapeutics, Pharmacology, and Microscopy)*: Stansell, L. W., Maidstone. Thorp, W., B.Sc. (Lond.), Limerick. Both for the Fellowship.

The examiners in Chemistry were Dr. Bernard Dyer, F.I.C., and Professor Percy F. Frankland, F.R.S., F.I.C., and the examiner in Therapeutics, Pharmacology, and Microscopy was Dr. Thomas Stevenson, F.R.C.P.

SALE OF FOOD AND DRUGS ACT.

(Reprinted from the "Times" of February 1, 1900.)

THE Right. Hon. W. H. Long, M.P., President of the Board of Agriculture, has appointed a departmental committee to inquire and report as to what regulations, if any, may with advantage be made by the Board of Agriculture under section 4 of the Sale of Food and Drugs Act, 1899, for determining what deficiency in any of the normal constituents of genuine milk or cream, or what addition of extraneous matter or proportion of water, in any sample of milk (including condensed milk) or cream shall, for the purposes of the Sale of Foods and Drugs Acts, 1875 to 1899, raise a presumption, until the contrary is proved, that the milk or cream is not genuine.

The committee will consist of the following gentlemen, viz., Lord Wenlock, G.C.S.I., G.C.I.E. (chairman), Mr. George Barham, Mr. George Cowan, Major Patrick George Craigie (an assistant secretary of the Board of Agriculture), Mr. S. W. Farmer, Mr. Shirley F. Murphy, M.D., Prof. Thorpe, F.R.S. (Principal Chemist of the Government Laboratories), and Mr. J. Augustus Voelcker, Ph.D. Mr. Robert Henry Rew, of the Board of Agriculture, will act as secretary to the committee.

THE ANALYST.

MARCH, 1900.

PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS.

THE monthly meeting of the Society was held on Wednesday evening, February 7, in the Chemical Society's Rooms, Burlington House. Mr. Alfred H. Allen, in the absence of the President, occupied the chair.

The minutes of the annual general meeting were read and confirmed.

Certificates of proposal for election to membership in favour of Messrs. Norman Parr Booth, John Macallan, and C. T. Tyrer were read for the second time; and certificates in favour of Messrs. James Eugene Kirkpatrick, Analytical Chemist, Taunton; A. J. Murphy, Analytical Chemist, 11, Lyddon Terrace, Leeds; and John Alexander Ramsay, Chief Chemist and Manager, Sugar Experiment Station, Mackay, Queensland, were read for the first time.

Mr. B. W. J. Warren was elected a member of the Society.

The following papers were read: "Note on the Separation of Oleic Acid from other Fatty Acids," by J. Lewkowitsch, Ph.D.; "Analysis of a Sample of Treacle and of a Sample of so-called 'Golden Syrup,'" by C. G. Matthews and A. Hyde Parker; and a "Note on Sour Milk," by H. Droop Richmond and J. B. P. Harrison.

THE PRESIDENT'S ANNUAL ADDRESS.

(Delivered at the Annual Meeting, January 24, 1900.)

IN addressing the Society at the close of a year of office, it is the duty of your President to take stock of the present condition of the affairs of the Society, to review the proceedings of the year, not without remembrance of older histories, and finally to forecast with "intelligent anticipation" the future course of events. The past we may regard with mixed feelings when we consider hopes not fulfilled or realized, but we may still look to a future without a dark cloud in prospect.

Looking back upon the Presidents' addresses in past years, say for ten years, I find certain topics uppermost in the minds of former occupants of this chair—the position of the Society; the prospects of amendment of the laws relating to our official duties; and the relations of Public Analysts to what was known as Somerset House—and these appear still to be the points of chief interest at the present moment.

During the past year we have had to lament the loss of one of our Senior Honorary Fellows, Sir Edward Frankland, whose death last August came so unex-

pectedly, and is felt by many as a personal loss of no ordinary kind. Some here present doubtless owe to him no small part of their education as chemists, others have been associated with him in the affairs of the Royal Society, the Chemical Society, and especially in the Institute of Chemistry, with the affairs of which he was so deeply concerned, and of which he was the first President, and in the welfare of which he remained faithfully and constantly interested. Apart from his eminence in pure science, he held a position of enormous responsibility as a professional chemist, and the high standards of purity by which he judged the character of the water-supply of London have been, without question, largely instrumental in bringing that supply to its present excellent condition, and incidentally set a similarly high standard for other public supplies, not only in this country, but in Europe and the United States, and practically the whole civilized world. The Sixth Report of the Rivers Pollution Commission, that monument of industry and research, in which his share was so large, co-ordinated and established on a sound basis the true principles for the scientific judgment of waters—their comparison with natural supplies of the districts where they are obtained.

An old member of our Society, Dr. Hodges of Belfast, has passed away at the ripe age of eighty-four, and will be much missed in the North of Ireland, where for many years he had done such good work, of which a notice recently appeared in THE ANALYST.

Two other senior members and veteran chemists have been lost to us—Mr. G. H. Ogston, long in professional practice in London, and Mr. R. Oxland, of Bristol, one of the earlier Public Analysts, and who was appointed in 1878 at Plymouth and Davenport.

As our Society was founded at the end of 1874, and held its first general meeting on February 25, 1875, it is now a quarter of a century old, half-way to its jubilee—at the silver-wedding age—and I am happy to say the Society was never stronger numerically or financially, and, I think I may fairly add, never more efficient or vigorous. The new rules came into operation at the beginning of 1899, and although a few associates gave up their connection with us, out of the total number of thirty-two no less than fifteen were elected at the first possible meeting, and eight others have since taken up their freedom, thus increasing the strength of the Society and bringing the younger members into closer association with its business. Forty-one members in all have been elected, making our actual members 9 honorary and 248 ordinary members—a complete total of 257. Two members have withdrawn during the year, but I think, owing to the judicious changes in our rules during the office of my predecessor (Dr. B. Dyer), we may look forward to yet further development.

The Honorary Treasurer has, I am happy to say, been able to give us a cheerful report of his exchequer, notwithstanding the fact of an exceptional amount of expenditure upon the journal and for printing expenses in connection with the Food and Drugs legislation of the past year. I am sure we are most grateful to him for the trouble he has taken on our behalf, not only by those gentle reminders which I hear he has sometimes to send to the forgetful few, but for the zeal he shows in the scrutiny of the various expenditures which are in his departmental control. One

way in which we may venture to show our gratitude is by sending our annual guinea without waiting for the gentle reminder.

I think we all feel that THE ANALYST is year by year increasing in interest and usefulness, and this is dependent upon those gentlemen who contribute their original and valuable work to its pages, and also to our friend Dr. Sykes, the Editor, who, with his coadjutors gather and condense so admirably the analytical matter constituting the rest of the journal. Possibly before long some enlargement of THE ANALYST may be necessary if the number of original contributions increases; but this will not necessarily mean loss to the Society, as growth of this character will be covered by an increase of membership and increased sales.

THE ANALYST is, of course, one of our heaviest items of expenditure, but at the same time one of the most necessary, and keeps the Society alive: it is the bond of union between those present at the meetings and absent members, which no Home Ruler would wish to repeal. The Society, however, has been urged again recently to modify the character of THE ANALYST, and save some part of the expense of its maintenance by accepting advertisements which are at present excluded, relating to articles of food and drink, as well as medical preparations of various kinds. We have not acceded to the suggestions, and I believe we have acted in the best interests of the Society and of professional chemistry. Unless control were exercised, some of the most objectionable forms of advertisement, with their testimonials of a similar class, would appear in the pages of our Journal, while, if a censorship were established, by which one manufacturer's advertisements might be excluded and another admitted, the fact would be taken as a form of guarantee of the wares, or an implied acquiescence in the terms of the advertisement. As Public Analysts, we must feel we are in a delicate position in this regard, and we have acted, therefore, on the simple intelligible principle of inserting no advertisements relating to articles which may be within the purview of the Food and Drugs Acts, and which we may be called upon to examine officially. This policy will, I believe, commend itself to the members of the Society generally, and will, I trust, be adhered to by our uccessors who may be responsible in the future for the conduct of the Society's affairs.

The list of papers read before the Society numbers twenty-eight, and some of the contributions are of permanent value and importance, and much interesting discussion on some of the papers has taken place at the evening meetings. Now that the amended and extended definition of food has been brought into the Act, a very large number of additional articles, and especially sauces and condiments, will come before the notice of analysts, and I hope to see during the next year further contributions to our knowledge from a study of the composition of such articles, now that more attention has been given to them. Those younger members anxious to find fresh ground for research need not want for material for some time to come.

Papers read in 1899:

"The Occurrence of Barium Compounds in an Artesian Well Water." By John White.

"A New Test for Formaldehyde." By Norman Leonard, B.Sc., and Harry M. Smith.

"On Caper Tea." By Charles Estcourt.
"The Adulteration of Sweet Spirit of Nitre with Potassium Nitrate." By W. F. Lowe, A.R.S.M.
"Some Analyses of Ginger." By E. G. Clayton.
"Note on Boric Acid in Milk Samples." By E. G. Clayton.
"Caper Tea." By John White.
"Note on a Possible Source of Error in Modifications of the Leffmann and Beam Method of Fat Estimation in Milk." By H. Droop Richmond and F. R. O'Shaughnessy.
"On a Recent Ginger Case." By E. J. Bevan, Bernard Dyer, D.Sc., and Otto Hehner.
"The Composition of Milk and Milk Products." By H. Droop Richmond.
"An Attempt to Compute Mathematically the most Probable Limits for Milk." By H. Droop Richmond.
"Note on the Estimation of Ether and Alcohol when mixed with Petroleum Ether and other Solvents." By H. Droop Richmond.
"The Estimation of Fat in Milk, using Petroleum Ether as a Solvent." By H. Droop Richmond and C. H. Rosier.
"Notes on Milk Analysis, including the Molybdate Test for Cane-sugar." By L. de Koningh.
"A New Form of Distilling Flask for Use in Water Analysis." By Mr. W. P. Skertchley.
"The Assay of Belladonna, B.P." By F. C. J. Bird.
"On the Use of Boric Acid and Formaldehyde as Milk Preservatives." By S. Rideal, D.Sc., and A. G. R. Foulerton, F.R.C.S.
"The Value of the Estimation of Pentosans in Food Materials." By Otto Hehner and W. P. Skertchly.
"On some Comparative Analyses of, and Digestive Experiments with, White and Wholemeal Breads." By Otto Rosenheim, Ph.D., and P. Schidrowitz, Ph.D.
"Note on Coffee Extracts." By C. G. Moor, M.A., and Martin Priest.
"On the Determination of the Iodine Value." By J. Lewkowitsch, Ph.D.
"On the Influence of Ammonium Salts on the Precipitation of Nickel by Ammonia." By Arthur Marshall.
"The Meaning of the Acetyl Value in Fat Analysis." By J. Lewkowitsch, Ph.D.
"On Some Analyses of Modern Dry Champagne." By Otto Rosenheim, Ph.D., and P. Schidrowitz, Ph.D.
"Note on Asafœtida." By J. M. Martin, B.A., M.B., and C. G. Moor, M.A.
"On the Determination of the Iodine Value." By Dr. J. J. A. Wijs.
"Treacle or Golden Syrup." By E. W. T. Jones.
"On a Method for Distinguishing between Hops and Quassia." By Alfred C. Chapman.

The prospects of amendment of the Food and Drugs Act of 1875—the Act which brought us and our Society into being—have occupied the Society and its officers for several years; but, with all its admitted defects, the old Act has worked

to the advantage of the public in no small degree. Mr. Hehner, in his address as President in 1892, pointed out that the percentage of adulteration, which from 1877-81 was 16·2 per cent. of adulterated samples, had fallen to 11·2 per cent. in 1890. The total number then analysed in the year was 27,465, of which 3,069 were adulterated; and it is interesting to note that the latest accessible return (1898) gives 49,555 samples examined, with 4,319 returned as adulterated—a proportion of 8·7 per cent. The Act has done much to check adulteration, although in ten County Councils and sixteen boroughs its provisions have been greatly or altogether neglected.

In the dark ages of 1890 Mr. Hehner observed that Oxfordshire was at the bottom of the list, but I am glad to say we now occupy a less conspicuous position.

In 1893 Mr. Hehner reviewed the Bill of Dr. Cameron which had met with the doom of the Innocents, and was not lamented; but that year was important for a discussion of proposals in this Society for amending the Acts, and the suggested Chemical Department of the Local Government Board. Our President in 1896 urged the need of the establishment of standards and limits, to be fixed by a competent and authoritative body on which Public Analysts should be represented; the Adulteration of Food Committee sat and reported, and, as you know, embodied in their report many of the suggestions and proposals of our Society.

The session of 1897-98 saw the introduction of another attempt justly described by the President, Dr. Dyer, as disappointing, since it almost ignored all the improvements, so much needed, which had been recommended by the Committee of the House after an exhaustive and careful inquiry. Our Council, as you will remember, took up a line of strong opposition, and the Bill was happily dropped.

At length, in 1899, the new Food and Drugs Bill was brought in, and, being a serious attempt to carry out the recommendations of the special committee, was on the whole favourably received; and after much interesting discussion and considerable changes, passed through Parliament, and came into operation in January of this year, 1900. The Society has been already informed of the modifications suggested by your Council, and the courteous way in which they were considered and received, and I am pleased to say in many cases the suggestions were accepted by Mr. Long and are incorporated in the Act.

Many of the new proposals emanated from the committee of 1896, and were commented upon by your President two years since. Experience alone can teach us the defects of this Act, which will indeed be a marvel of perfection if everybody is satisfied with it. The buyer and the seller, the inspector and the analyst, the prosecuting and defending solicitors, the local authorities, the Board of Agriculture, the Government chemists, besides the magistrates and Her Majesty's judges, will be concerned in various ways in carrying out, interpreting, or finding loopholes in the provisions of the new Act, and its effects will not perhaps be quite in accordance with what was intended; but I look upon many of the new provisions as excellent, and have great hopes of their future usefulness.

It is probable that the number of samples purchased under the Act will increase, especially in certain districts, where little use has been made of its provisions in the past, now the machinery can be set in motion from the Central Authority, and the provisions referring to the use of warranty as a defence will, it is to be hoped,

protect the innocent retailer, and enable the real offender to be punished for adulteration, in fact, put the saddle on the right back.

An important new departure arises from the provision that a certificate of analysis by a Public Analyst, made for the defendant, will be evidence of the facts stated therein, without his personal attendance at the hearing. This seems a fair arrangement, and will be a convenience to those gentlemen who may be asked to make an analysis for the seller. Some fears have been expressed that this provision may lead to undignified disputes in court, and that contradictory certificates will be produced, to the detriment of our profession and the hindrance of justice, but I do not share in these misgivings. Every Public Analyst who may be asked to examine an article for the defendant will do the work with the full responsibility which rests upon him for all his official analyses, and I have no doubt will report impartially on the case. And if he should conscientiously differ in opinion from the analyst for the other side, the case would probably be referred to the Government Laboratory to decide between the opposed opinions. It must be remembered that the correctness of a Public Analyst's certificate is seldom seriously disputed; the number of cases referred to the Government Laboratories is a very small proportion of those reported as adulterated (about 1·5 per cent.), and in three cases out of four the analyst's certificate is confirmed by the referees.* It will doubtless happen that a defendant will have his reserve sample examined, not possibly knowing its real character, only to find the certificate of the buyer confirmed in the large proportion of cases.

Another consequence arising out of the new Act is the appointment of a Departmental Committee to inquire into the questions of preservatives and colouring matters in food. That Committee is now sitting and taking evidence, and, as is only natural where diverse interests are concerned, evidence of very conflicting character has been tendered. But there appears to be a growing conviction that the unrestricted use of preservatives, which hitherto it has been difficult to deal with, should not go on, and some limits should be imposed, and for some articles an entire prohibition of preservatives is necessary. Your Society appointed two representatives, the President and Dr. Stevenson, and many members of the Society have individually given, or will give, their experiences and opinions to the Committee; others, in response to an invitation sent out, have furnished your representatives with much valuable testimony as to the nature and extent to which preservatives are employed in different parts of the country. I anticipate, when the Committee reports, that many disputed points as to the desirability or otherwise of permitting certain additions to food will be finally settled and disposed of.

It may be some time before the Committee is able to collect all the information they would desire, and I should like to invite any of our members who have not yet returned the schedules of preservatives and colouring matters to send them to the secretaries, with any information they may be able to gather on the subject. Probably some suggestions to inspectors in various districts may lead to the purchase of articles on which our information is scanty, as they are seldom bought under the Act.

* 49,555 samples in 1898; 4,319 adulterated, 74 references, with only 17 disagreements.

Our relations with Foreign Powers, I am happy to say, continue friendly, although your Council has had some diplomatic correspondence with the Government chemist, which appeared in the journal of the Society. I do not desire to revive old controversies, and I am glad to notice that the proportion of cases in which the opinion of the referees differed from that of the Public Analyst in respect of disputed samples is diminishing. Doubtless there will be some differences of opinion in the future, as in the past, since the natural variations in composition of animal and vegetable products makes it impossible to fix an exact limit or analytical standard; but since some standard must be used in forming a judgment after every analysis, it is surely better to settle by friendly discussion the lines on which we will work and the limits of variation to be allowed. In this way the unsatisfactory conflict of evidence between a responsible public officer and a Government official could frequently be avoided.

In the report of the Government Laboratory on the samples referred under the Food and Drugs Act, I notice that seventy-four samples came before them for judgment, of which forty-seven were milks. They confirmed the Public Analyst's opinion as to forty milks, and dissented from him in seven cases, which looks as though we have somehow arrived at a common standard of reference for the composition of milks. Also, in three cases out of four, the analyst's verdict was confirmed as to undue dilution of spirits, and here, again, the standard is very definite. But in those cases of ground ginger and caper tea referred to in our correspondence with the Government chemists it is evident that the divergence of opinion rests solely on the diverse interpretation of practically concordant analyses; how far ginger may be impoverished by washing, and how much extraneous matter should be permitted in tea, are the simple questions at issue, and these should be settled once and for all.

The President (Dr. Stevenson) addressing the Society in 1897 quoted the statement of the Adulteration Committee, that "they regarded it as of great importance, in the interests of the public, that Public Analysts should, as far as possible, be made acquainted with the methods adopted by the Government Laboratory in the analysis of food, and with the considerations kept in view by them in determining whether an article has been adulterated"; and added the comment that "this pronouncement we may expect to have a generous and not tardy response, and that the official book, which ought to be, and possibly is, in preparation by Somerset House, placing all the analytical methods used in food and drug analysis, and the deductions that ought to be drawn from the results of analysis, at our disposal, will be welcomed by Public Analysts as a priceless boon."

Dr. Thorpe, at the Society's Dinner in 1896, told us "he had no stronger desire than that both he and we, public servants as we are, should work together cordially and harmoniously in the discharge of our public duties," and he looked forward to an opportunity of personal adjustment of such questions as had been referred to.

Well, gentlemen, I am extremely pleased to say that the Chief Chemist has recently made a suggestion for a conference between this Society and some of the staff, with the special view of coming to an understanding upon the methods to be employed in judging samples of margarine under the new Act. This proposal, naturally, has been cordially accepted, and I have every confidence that we shall

arrive at an understanding both as to a common method of working and the analytical limits to be adopted whereby we can obtain concordant results when the samples have to be examined by both sides.

I venture also to hope that this is only the first conference of its kind, which will be followed by others relating to points where any differences of opinion may arise in regard to other articles.

NOTE ON THE SEPARATION OF OLEIC ACID FROM OTHER FATTY ACIDS.

By J. Lewkowitsch, Ph.D.

(*Read at the Meeting, February* 7, 1900.)

In the course of a technical research on oleic acid, I had occasion to examine two methods which had been proposed recently by Twitchell and by Farnsteiner—the one for the separation of oleic acid (and other liquid acids) from solid fatty acids, the other for the separation of oleic acid from less saturated fatty acids.

I find the two methods practically useless for quantitative purposes, and, unpleasant though it be to publish results of this kind, I think some little service may be rendered to my colleagues by placing my results on record.

Twitchell's method is based on the insolubility in petroleum ether of the oleo-sulpho compound, obtained on treating a mixture of saturated and oleic acids with 85 per cent. sulphuric acid. He obtained apparently very good results in the case of a commercial oleic acid of a cold test of 12° C., his method yielding a little over 7 per cent. solid acids.

I examined a commercial oleic acid made from tallow, having the solidifying-point of 8·5° C. and the iodine value 82·27, so that it may be accepted that the acid contained about 90 per cent. of true oleic acid, and practically very little of less saturated acids, if any.

I obtained in two experiments, when the petroleum ether was allowed to stand with the compound for a short time only, from 21·6 to 25 per cent. solid acids, and in two other experiments, where the mass was allowed to stand with the petroleum ether overnight, from 17·8 to 18·7 per cent. solid acids.

Another sample of oleic acid from the same source, having the iodine value 84·16 and hence containing about 7·25 per cent. solid fatty acids, gave by Twitchell's method from 35·36 to 44·30 per cent. solid acids. The best results were obtained by cooling the oleic acid in ice-water and allowing to stand with the acid for twelve hours before adding the petroleum ether. But even then, in four experiments, from 15·35 to 16·58 per cent. of soluble (supposed solid) acids were found, so that further attempts had to be abandoned.

Farnsteiner's method is based on the insolubility of barium oleate in cold benzene containing a little alcohol, in which menstruum the barium salts of the saturated acids are also insoluble. If this method yielded satisfactory results, an important step in the resolution of mixed fatty acids containing acids of the series $C_nH_{2n}O_2$, $C_nH_{2n-2}O_2$,

$C_nH_{2n-4}O_2$, and less saturated would be made, as all the acids of greater unsaturation than oleic acid could be removed by means of crystallizing the barium salts from benzene containing the prescribed amount of alcohol. From the mixture of saturated acids and oleic acid then remaining, the oleic acid could be separated in a practically satisfactory manner by well-known methods.

I therefore examined samples of linseed, cottonseed, and olive oils. The result is set out in the table. The discordant results suggested the examination of oleic acid. The oleic acid of iodine value 84·16 was employed for the experiments. A glance at the table shows the hopelessness of the method.

TABLE I.

Results of Farnsteiner's Method.

	Amount of Fatty Acids.				
	1st Filtrate.	2nd Filtrate.	3rd Filtrate.	Insoluble.	Total found.
Linseed oil	90·30	4·71	1·57	2·54	99·12
Linseed oil	86·38	4·55	0·98	2·43	94·34
Linseed fatty acids	61·62	17·20	7·80	1·50	88·12
Linseed fatty acids	65·32	14·02	4·40	2·80	86·54
Olive oil	28·20	26·65	10·67	24·16	89·68
Olive oil	29·17	18·65	29·30	12·12	89·77
Olive oil	33·58	22·55	18·36	11·53	86·02
Olive oil	31·93	21·64	24·31	12·34	90·22
Cottonseed oil	64·36	9·58	3·43	18·86	96·23
Cottonseed oil	61·44	10·57	3·00	19·95	94·96
Cottonseed oil	44·25	9·81	8·80	24·52	87·38
Cottonseed oil	44·67	12·26	7·80	22·26	86·99
Oleic acid	85·94	8·05	3·00	2·94	99·93
Oleic acid	85·96	5·28	2·68	3·49	97·41
Oleic acid	42·84	37·74	20·18	—	100·76
Oleic acid	23·28	43·74	29·43	4·20	100·65
Oleic acid	23·70	67·37	5·96	0·44	97·47
Oleic acid	10·68	66·26	20·12	0·40	97·46
Oleic acid	53·73	12·51	29·24	4·70	100·58
Oleic acid	47·90	15·00	34·45	0·63	99·95

An explanation of my failure is found in the following table, which proves that, contrary to Farnsteiner's statement, barium oleate is not insoluble in benzene, and much less so in the proposed mixture of benzene and alcohol.

TABLE II.

Barium Oleate, boiled 1½ hours under reflux condenser with 50 c.c. of Benzene, containing—

95 per Cent. Alcohol.		Yielded Soluble Salt.	
Cubic cents.	0·0	Per cent.	5.3
,,	0·0		14·4
,,	0·5		31·34
,,	1·0		37·17
,,	2·0		57·34
,,	2·0		56·59
,,	2·5		59·94

The experiments set out in the above table were carried out by Mr. C. D. Robertshaw, to whom my best thanks are due.

DISCUSSION.

Mr. HEHNER said that, on heating oleic acid with potassium hydroxide, one molecule of hydrogen was given off, whereas in the case of stearic acid and palmitic acid there was no evolution of hydrogen. It was to be presumed, also, that in the case of linolic acid, which contained one molecule of hydrogen less than oleic acid, no hydrogen would be given off on heating with potassium hydroxide. It would therefore seem that the hydrogen evolved on heating the mixture of fatty acids with potassium hydroxide ought to bear some relation to the quantity of oleic acid present, and might afford a means of determining it.

Mr. C. A. MITCHELL inquired whether the author had made any experiments on the lines of another paper of Farnsteiner, in which lead elaïdate was said to behave in petroleum spirit like the lead salts of the saturated fatty acids, though the results then obtained were not sufficiently constant for quantitative purposes. F. G. Edmed, in a recent paper before the Chemical Society, stated it to be possible, under certain circumstances, to convert oleic acid into elaïdic acid quantitatively, so that Farnsteiner's results in this direction, provided that they were reliable, might possibly now be of assistance in solving the problem.

Dr. LEWKOWITSCH said that Mr. Hehner's suggestion was certainly a valuable one, although the evolution of hydrogen could not be expected to be strictly quantitative so as to determine thereby pure oleic acid; and it would have to be shown that no evolution of hydrogen took place in the case of linolic acid. In reply to Mr. Mitchell, his own attempts to prepare elaïdic acid on a considerable scale led him to think it hardly possible that Edmed could have really obtained, as he stated, 98 per cent. of pure elaïdic acid. Also Farnsteiner had tried to convert quantitatively oleic acid into elaïdic, but had failed.

THE RELATION BETWEEN SPECIFIC GRAVITY, FAT, AND SOLIDS-NOT-FAT IN MILK.

By Norman Leonard, B.Sc., F.I.C.

Many of the formulæ which have at various times been devised to express the relation between the specific gravity, fat, and solids-not-fat in milk are open to criticism on one or more of the following points. In the earlier investigations the number of samples analysed was too small to allow of accurate conclusions being drawn, and the fat was assumed to exercise an influence on the specific gravity corresponding with its density in the isolated state. In many cases the analyses appear to have been made within a comparatively short period, a point which is of importance in connection with the fact, pointed out by Richmond (Analyst, xix., 73-87; xx., 1-3), and confirmed by myself (*vide infra*), that there exists a periodical variation in the constitution of milk. It would seem, also, that the formulæ hitherto proposed have been based on analyses executed by the most accurate, but not always the most generally used, methods, and under conditions more or less removed from those under which the practical analyst, for whose use the formulæ are intended, is compelled to work. The following remarks have reference to 137 samples of milk—genuine and adulterated—received throughout the year 1898 for analysis under the Sale of Food and Drugs Act; and although the analyses have no claim to absolute scientific accuracy, yet the deductions drawn therefrom may have a comparative and a confirmatory value, and the results will be of interest as being the outcome of ordinary routine work. As regards the analytical methods employed, the specific gravity was determined by a hydrometer, due corrections being applied for temperature and for inaccuracies in the graduation of the instrument; the total solids by evaporating 5 grammes to dryness in a platinum dish and drying the residue at 100° C. for three to four hours; the fat by the Werner-Schmid process; and the solids-not-fat by difference. Of the 137 samples examined 4 were "separated" milks, 74 contained from 1 to 3 per cent. of butter-fat, and 9 contained over 4 per cent. of fat; probably the majority of the samples were watered, since 105 contained less than 8·5 per cent. of solids-not-fat. The average composition was: specific gravity, 1029·3; total solids, 11·05; fat, 2·94; solids-not-fat, 8·11 per cent.

Assuming the excess gravity (G), fat (F), and solids-not-fat (S) to be connected by the equation $G = Ss - Ff$, where s and f represent the number of units by which the specific gravity (water = 1000) is raised or lowered by each 1 per cent. of solids-not-fat and fat respectively, the most probable values of s and f are given, according to the method of least squares, by the formulæ

$$s = \frac{\Sigma(SG).\Sigma(F^2) - \Sigma(FG).\Sigma(SF)}{\Sigma(S^2).\Sigma(F^2) - [\Sigma(SF)]^2},$$

$$f = \frac{\Sigma(SG).\Sigma(SF) - \Sigma(FG).\Sigma(S^2)}{\Sigma(S^2).\Sigma(F^2) - [\Sigma(SF)]^2}.$$

Making the calculations indicated, the formula $G = 3{\cdot}905S - 0{\cdot}815F$ is obtained, whence, since T (total solids) = S + F, $F = 0{\cdot}827T - 0{\cdot}212G$. On comparing the fats calculated by this formula with the experimental values, it is found that the errors

lie between $-0{\cdot}33$ and $+0{\cdot}35$ as extremes; in 70 per cent. of the analyses the error is not greater than 0·1, and in 90 per cent. it is not greater than 0·2; the probable error is $\pm 0{\cdot}078$. As has been shown by Richmond (ANALYST, xviii., 169-171), the magnitude of the error appears to be independent of the amount of fat, but there is distinct evidence that the fat calculated exceeds that found as the solids-not-fat rise. The magnitude of the error also varies with the season, as is shown in the table below, in which the 137 samples, arranged in order of date, are divided into six batches of 20 and one of 17, and the average error calculated for each batch. This point will be again referred to later.

Numbers.	Average Error.	s.	f.
1 – 20	– 0·13	4·035	0·954
21 – 40	– 0·04	3·888	0·716
41 – 60	+ 0·03	3·933	0·975
61 – 80	+ 0·05	3·838	0·710
81 – 100	+ 0·07	3·786	0·650
101 – 120	– 0·01	3·941	0·905
121 – 137	– 0·04	3·884	0·720

It may be here pointed out that although 3·905 and 0·815 are respectively the most probable values for s and f in the equation $G = Ss - Ff$ connecting specific gravity, fat, and solids-not-fat, the constants in the equation $F = 0{\cdot}827T - 0{\cdot}212G$, deduced therefrom by means of the relation $T = S + F$, are not the most probable for an equation connecting F, T, and G. The values of a and b in the equation $F = aT - bG$, independently calculated by the method of least squares, are 0·809 and 0·205 respectively; but the results obtained by the use of these figures are practically identical with those given by the formula $F = 0{\cdot}827T - 0{\cdot}212G$, and the probable error is not appreciably less.

In order to ascertain whether the changes in the error at different times of the year are due to variations in the value of either s or f, or of both these factors, their values were independently calculated for each of the batches in the table given above. It will be seen that both s and f vary in a somewhat irregular manner, but that at the time when the error attains its greatest positive value both s and f are at a minimum, whilst the largest negative error coincides with the maximum values of s and (nearly) of f.

The secular variation of the error suggested the use of an equation of the type $F = aT - bG + K$, in which K might be varied with the time of year. In this case the constants a, b, and K must be so chosen as to fulfil the condition

$$\Sigma(aT - bG + K - F)^2 = \text{a minimum.}$$

The values of K calculated for the seven batches of samples separately are very small—less than 0·002—but taking the whole number of samples, the equation $F = 0{\cdot}796T - 0{\cdot}210G + 0{\cdot}30$ is obtained. This gives results with a probable error of $\pm 0{\cdot}076$, and is therefore only slightly more accurate than either of the two formulæ previously suggested. On comparing the fats calculated by this formula with the experimental values and plotting the average monthly errors against the months of the year, a rough curve is obtained having a minimum point in February and a

maximum in August. The following average errors were read from the smoothed curve:

Month.	Error.	Month.	Error.
January ...	−0·12	July ...	+0·11
February ...	−0·13	August ...	+0·12
March ...	−0·09	September	+0·11
April ...	−0·03	October ...	+0·06
May ...	+0·02	November...	−0·01
June ...	+0·07	December...	−0·07

The corresponding corrections, which are of opposite sign to the errors, may be applied to the results obtained with the formula $F = 0{\cdot}796T - 0{\cdot}21G + 0{\cdot}30$, or, what is the same thing, the correction may be applied to the constant K, which thus assumes a different value for each month. With the formula thus corrected results are obtained with a probable error of $\pm 0{\cdot}065$. In 75 per cent. of the analyses the error is not greater than 0·1, and in 94 per cent. it does not exceed 0·2.

The equation $F = 0{\cdot}796T - 0{\cdot}21G + 0{\cdot}30$ may be written

$$F = \tfrac{4}{5}(T - \tfrac{G}{4}) + (0{\cdot}3 - 0{\cdot}004T - 0{\cdot}01G).$$

The value of the term within brackets practically never exceeds $\pm 0{\cdot}1$, and in the vast majority of milks dealt with by Public Analysts it is considerably less; hence the simple formula $F = \frac{4}{5}(T - \frac{G}{4})$ corresponds fairly accurately with the results.

The fats calculated in accordance with the formula $T = \frac{G}{4} + \frac{5}{4}F + 0{\cdot}14$, proposed by Richmond in 1895 (ANALYST, xx., 57), are higher than those given by the formula $F = 0{\cdot}796T - 0{\cdot}21G + 0{\cdot}30$ in the case of milks containing over 10 per cent. of total solids, and lower when the solids are less than 10 per cent. The difference does not, however, exceed 0·1, except in the case of rich milks.

My thanks are due to Dr. Thomas Stevenson for permission to make use of results obtained in his laboratory.

ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

FOODS AND DRUGS ANALYSIS.

Determination of Glucose in Treacle. A. K. Miller and J. P. Potts. (*Journ. Soc. Chem. Ind.*, 1899, 1091.)—The authors are of opinion that as yet no satisfactory method has been devised for the quantitative analysis of sugar syrups adulterated with glucose, and that, failing this, analysts must be contented with a qualitative test. This, the authors consider, is afforded by the polarimeter; in many cases a single observation by this instrument will show the presence of glucose. It is in general better to extend the examination and proceed as follows: Determine reading of a 10 per cent. solution of the syrup; invert the cane sugar in a portion of the solution, again determine rotation, and calculate the rotation due to cane sugar. If this be deducted from the original rotation, the difference will represent the rotation due to

dextrose and lævulose if the sugar is a genuine sugar syrup, and the result will be a lævo-rotation. As dextrose and lævulose are not invariably present in equal amounts, no suspicion is to be attached to a sample showing a slight dextro-rotation at this point; but if glucose is present, a marked dextro-rotation will be found, and its degree will afford some guide as to the amount of glucose added. Should any doubt still remain, the sugar may be fermented, and the rotation of the solution again determined. As glucose syrups always contain large amounts of dextrin, the rotatory power of the fermented solution affords a good confirmatory test. A table of the analyses of various syrups, pure and adulterated, bearing out the authors' contentions, is appended. W. J. S.

On the Influence of Feeding on some of the Constants of Butter. A. Ruffin. (*Ann. de Chim. Anal.*, 1899, iv., 383-385.)—In order to determine to what extent the nature of the food given to the cow influences the constants of the butter, the author has analysed a number of samples, and summarizes his results in the following table:

Food of Cows.		Refraction No.	Saponification Value.	Reichert-Wollny No.
Fodder, lucerne, etc.	Max.	33	232	34·9
	Min.	30	224	27·6
Cotton-seed oilcake	Max.	30	228	29
	Min.	28·5	222	26·4
Cotton-seed oilcake and fodder	Max.	30	229	30
	Min.	29·5	221	28·4
Earthnut oilcake	Max.	31	228	29
	Min.	30	225	26·9
Earthnut oilcake and fodder	Max.	30	229	32
	Min.	28	221·9	28
Cocoanut oilcake	Max.	33	240	31
	Min.	32	231	28·5

C. A. M.

A New Reagent for Alkaloids, especially Opium Bases. Mecke. (*Zeits. öffentl. Chem.*, 1899, v., 351; through *Chem. Zeit. Rep.*, 1899, 273.)—A 0·5 per cent. solution of selenious acid in strong sulphuric acid gives characteristic colour reactions with colchicine, digitalin, veratrine, and more particularly with the alkaloids of opium. The reagent strikes a conspicuous green tint even with 0·005 milligramme of morphine and codeine, and the reaction is therefore more delicate than its predecessors, appearing immediately and in the cold. When the colours produced by sulphuric acid alone are red, they are very liable to be masked by the brown shade formed by the action of the acid upon extractive matters if the alkaloids are impure; the green yielded by selenious acid in presence of codeine and morphine, on the other hand, is distinctly visible even if the solution turns brown. F. H. L.

TOXICOLOGICAL ANALYSIS.

Determination of Morphine in Toxicological Cases. Russwurm. (*Pharm. C. H.*, 1899, xl., 544; through *Chem. Zeit. Rep.*, 1899, 284.)—The author modifies the usual process slightly so as to recover the alkaloid in a crystalline condition. The original ammoniacal solution is extracted with warm amylic alcohol, the latter is shaken with hydrochloric acid, the aqueous liquid is supersaturated with ammonia, and the solution is extracted with chloroform. The chloroform is evaporated till only a few c.c. are left, mixed with 50 grammes of petroleum spirit, and set aside for twenty-four hours. The liquor is poured off, the morphine crystals are dried first in the air, then at 80° or 90° C., and finally weighed. F. H. L.

Estimation of Chloral Hydrate in Toxicological Work. Russwurm. (*Pharm. C. H.*, 1899, xl., 543; through *Chem. Zeit. Rep.*, 1899, 283.)—Chloral hydrate only distils with difficulty in a current of steam. The substance should be made alkaline and then distilled; a portion of the distillate is boiled with alcoholic potash under an inverted condenser to split off the chlorine, which is then determined as usual. F. H. L.

ORGANIC ANALYSIS.

The Chemical Identification of Vegetable Fibre. E. Jandrier. (*Ann. de Chim. anal.*, 1899, iv., 337, 338.)—This is based on the property possessed by cellulose of being converted by the action of sulphuric acid into carbohydrates of an aldehydic character, which give colour reactions with various phenols.

Thus, for example, to detect cotton-fibre in a woollen fabric, the sample is well washed and warmed for thirty minutes on the water-bath with sulphuric acid of 20° Bé. The liquid is then diluted and tested for carbohydrates in the following manner: About 0·01 gramme of a phenol, such as resorcinol, is placed in a test-tube, into which is also introduced 1 to 2 c.c. of the incomplete solution of the sample. A little sulphuric acid, which must be completely free from nitrous compounds, is then poured down the sides of the tube, which, if necessary, is slightly warmed in the water-bath.

If the liquid obtained by treating with sulphuric acid a quantity of the fabric containing 1 gramme of cotton be diluted to a litre, the colour of the product becomes orange-yellow with resorcinol; violet with α-naphthol, morphine, and codeine; green with gallic acid; brown with hydroquinone and pyrogallol; and rose with thymol, menthol, etc. (*Cf.* ANALYST, xxi., 295.)

These reactions are said to be so extremely sensitive that traces of cotton can be detected even in dyed fabrics, and it is rarely necessary to decolorize the solution with animal charcoal.

By comparing the colours thus obtained with those given by solutions derived from a known weight of cotton under identical conditions as to temperature, etc., the reaction may be made quantitative.

C. A. M.

The Estimation of Sugar in Diabetic Urine. G. Patein and E. Dufau. (*Journ. Pharm. Chim.*, 1899, x., 433-438.)—As certain albuminous substances in urine are incompletely precipitated by the basic or normal acetate of lead, the authors prefer a solution of mercuric nitrate for the purpose. This is prepared by mixing 200 c.c. of acid mercuric nitrate (*nitrate acide de mercure*) with 500 to 600 c.c. of water, adding a few drops of a solution of sodium hydroxide, until, on shaking, a slight yellow precipitate is formed, and making up to a litre.

In making an analysis, this reagent is added to 50 c.c. of the urine so long as a precipitate is formed. Sodium hydroxide solution is then added drop by drop with continual agitation until the liquid is very slightly alkaline, after which it is made up to 100 or 150 c.c. and filtered. The filtrate will contain only traces of mercury, and can be used directly for the volumetric determination of the sugar.

For a polarimetrical estimation, the last traces of mercury may be eliminated by adding a small quantity of sodium hypophosphite, and gently heating the liquid, when the mercury will be precipitated in the metallic form, and the filtrate can be examined in a polarimeter in the ordinary way.

In order to determine the extreme maximum limit at which it might be necessary to use the mercuric nitrate reagent, the authors added to a diabetic urine 10 grammes of peptones and 20 grammes of sodium chloride per litre, and 50 c.c. of this urine only required 60 c.c. of the reagent.

The following table is given in illustration of the author's experiments :

	Sugar, Grammes per litre.			
	1.	2.	3.	4.
Estimated by polarimeter :				
Urine, simply filtered	47·15	55·35		
,, lead subacetate precipitation ...	45·50	55·15	11·95	7·20
,, normal lead acetate precipitation	48·17	55·96	12·40	7·45
,, mercuric nitrate precipitation ...	48·20	55·50	12·71	7·79
,, with addition of peptones and salt, mercuric nitrate precipitation	47·90	55·60	12·30	7·38
Estimated with Fehling's solution in the urine after precipitation with mercuric nitrate	47·50	55·30	12·10	7·20

From these results the authors conclude : (1) That dextrose may be precipitated by basic lead acetate, which may be replaced with advantage by the normal acetate. (2) That certain lævorotatory substances in urine are not precipitated by either of the acetates, and that in such cases concordant results may be obtained by the optical or volumetric methods by the use of acid mercuric nitrate in the manner described above.

They have also made satisfactory estimations by using mercuric sulphate as a precipitating agent in the proportion of from 2 to 5 grammes to 100 c.c. of urine.

C. A. M.

The Solubility of Essential Oils in Sodium Salicylate.* M. Duyk. (*Bull. de l'Acad. belge royale ;* through *Journ. Pharm. Chim.*, 1899, x., 448-452.)—*Oil of Lemon.* —Two treatments with sodium salicylate solution (specific gravity, 1·24) are usually

* *Cf.* ANALYST, this vol., p. 12.

sufficient for the complete extraction of the soluble constituents. A specimen of lemon oil, with specific gravity 0·856 and rotatory power +62·7°, yielded 4·40 per cent. of soluble substances.

The soluble portion was a colourless oil with an odour of citral. Its specific gravity was 0·912, and its optical rotation +2·14°. It was soluble in 20 volumes of alcohol at 60° C., and combined with sodium bisulphite.

The insoluble portion was a colourless oil with a slight odour. Its specific gravity (0·851) and boiling-point (176° C.) agreed with those constants of limonene, but its optical rotation was different.

Eucalyptus Oil.—This contains variable quantities of eucalyptol, according to its origin. As this substance is the most important constituent, the author considers that preference ought always to be given to the oil which yields the largest proportion of soluble constituents to a concentrated salicylate solution.

Oil of Fennel.—This is almost entirely insoluble.

Geranium Oil.—The nature and amount of esters vary in the different species, the oil of geranium palmarosa being relatively very poor in them as compared with the French, Turkish, and African oils. The separation of these esters by means of sodium salicylate solution is never complete, owing to the solvent action of the large quantities of free alcohol present.

Indian or Palmarosa Geranium Oil (*Andropogon Schoenanthus*).—Specific gravity, 0·890; optically inactive; free geraniol, 94·6 per cent. This was completely soluble in 4 volumes of the solution with 1 of water. The product of the acetylation of the oil was completely insoluble.

African Geranium Oil (*Pelargonium*).—Density, 0·892; rotation = −9·2°; geranylic esters, 29·9 per cent. This was almost completely soluble in concentrated salicylate solution. On treatment with 4 volumes of salicylate solution and 1 volume of water, it yielded 7 per cent. of insoluble constituents, probably terpenes.

French Geranium Oil (*Pelargonium*).—Density, 0·900; geranylic esters, 19·60 per cent. Almost completely soluble in the concentrated salicylate solution. On treatment with a dilute solution (4 : 1), it yielded equal parts of soluble and insoluble constituents.

Oil of Cloves.—This contains a large proportion of a phenol (eugenol) and a small quantity of a sesquiterpene (caryophyllene). When shaken with dilute salicylate solution (4 : 1) the whole of the phenol is dissolved, while the sesquiterpene is left. According to the author, good clove oil should contain at least 90 per cent. of soluble constituents.

Essence of Cherry Laurel.—This is completely soluble in dilute salicylate solution.

Oil of Lemon Grass.—This oil is rich in citral, and has also been found to contain geraniol and methylheptenone. When treated with a mixture of 4 parts of the concentrated salicylate solution and 1·5 part of water, it dissolves to a large extent (up to 91 per cent.).

In the author's experiments the insoluble portion had a dark colour, a strong and disagreeable odour, and was strongly lævorotatory. The soluble portion had a strong odour of citral, and had the same density (0·898). Its optical rotation was −0·56°, citral being inactive. C. A. M.

Reactions of Bromine with Phenol and the Cresols, a Process for Calculating the Composition of Mixtures thereof. H. Ditz and F. Cedivoda. (*Zeits. angew. Chem.*, 1899, 873 and 897.)—If either *o*- or *p*-cresol, in alkaline solution, is mixed with a known excess of bromine dissolved in sodium hydroxide, and the liquid is acidified with 1 : 1 hydrochloric acid and agitated for one minute, on adding potassium iodide and titrating with thiosulphate, 2 atoms of bromine will be found to have been taken up by each molecule of the aromatic compound. If either phenol or *m*-cresol is treated similarly, 3 atoms of halogen are absorbed. On the other hand, if either *o*- or *p*-cresol in presence of the bromine and sodium hydroxide is allowed to stand for ten minutes after acidification with strong sulphuric acid, then shaken for five minutes, and the insoluble matter removed by filtration, on adding potassium iodide to the filtrate and titrating with thiosulphate, 3 atoms of bromine will be found to have combined with each molecule of the phenolic body. If either phenol or *m*-cresol is treated in the latter manner, 4 atoms of bromine are absorbed.

Depending on these reactions, it is possible, by suitable calculation, to separate a mixture of any three of the above-mentioned phenols into one which combines with the lower (higher) proportion of halogen, and into two which combine with the higher (lower) proportion of halogen; the results being quite satisfactory. And it is also feasible, though with less accuracy, to separate a mixture containing phenol and each of the isomeric cresols into phenol, *m*-cresol, and *o*- plus *p*-cresol. For the former purpose only one process of absorption is requisite; to attain the latter, both must be carried out. When all four phenolic bodies are present simultaneously, the method of calculation is as follows: a is the total weight of mixed phenols, x the true phenol, y the *o*- plus *p*-cresol, z the *m*-cresol, b the bromine taken up when hydrochloric acid is used, and c the halogen absorbed after treatment with strong sulphuric acid and filtration.

$$x + y + z = a \ldots . \qquad \ldots . \qquad (1).$$

$$\frac{3Br}{94{\cdot}06} \times x + \frac{2Br}{108{\cdot}08} \times y + \frac{3Br}{108{\cdot}08} \times z = b \ldots . \quad (2).$$

$$\frac{4Br}{94{\cdot}06} \times x + \frac{3Br}{108{\cdot}08} \times y + \frac{4Br}{108{\cdot}08} \times z = c \ldots . \quad (3).$$

In order to obtain the original phenols in a condition suitable for bromination, all hydrocarbons, fatty acids, and water must be removed. The authors bring them into ethereal solution, dry the extract with calcium chloride or sodium sulphate, and distil off the solvent. The last traces of ether are driven off by heating the mixture two or three times in a fractionating flask to almost 180° C., and then driving over the phenolic bodies themselves. Care must be taken that the excess of bromine is sufficient in all cases; and in the presence of *p*-cresol, when brominating with hydrochloric acid, the time of standing must not be prolonged.

F. H. L.

Volumetric Estimation of Orthocresol with Bromine. R. Clauser. (*Oesterr. Chem. Zeit.*, 1899, ii., 585.)—The publication of Ditz and Cedivoda's recent process for the separation of mixtures of phenolic bodies (see preceding

abstract) constrains the present author to place on record certain similar results he has himself obtained in the investigation of *o*-cresol.

As Benedikt has already shown, in contact with a large excess of bromine, phenol yields bromoxytribromophenol, which is fairly resistant to the action of water, alkalies, and acids, but gives ordinary tribromophenol on treatment with potassium iodide, according to the reaction :

$$C_6H_2Br_3OBr + 2KI + H_2SO_4 = C_6H_2Br_3OH + HBr + K_2SO_4 + I_2.$$

Clauser has therefore examined the behaviour of the analogous bromoxy-compound of *o*-cresol to see whether, and under what conditions, it is attacked by potassium iodide to regenerate the dibromo-*o*-cresol. He finds that the cresol solution which is treated with the bromine should have a concentration of 1 : 30,000 or 1 : 40,000 ; that too large an excess of bromine should be avoided, the best proportion being 6 atoms of bromine to 1 molecule of the cresol ; that directly the liquid becomes yellow—which occurs in about five minutes, and is due, not to free halogen, but to the formation of the bromoxy-compound—the potassium iodide should be added ; and that the liquid should stand at least half an hour before titration, which should be done in the cold. By attending to these points, *o*-cresol can be determined by the Koppeschaar process with a maximum error of +0·5 per cent. ; exactly 4 atoms of bromine being employed in the reaction per 1 molecule of cresol. Under the conditions of the analysis there is no danger of the iodine liberated from the iodide reacting with the aromatic molecule. F. H. L.

Valuation of Indigo. **R. Clauser.** (*Oesterr. Chem. Zeit.*, 1899, ii., 521.)—This is essentially an improvement upon Schneider's naphthalene process (ANALYST, 1895, xx., 211). According to its quality, 0·3 to 0·5 gramme of the indigo is mixed with 2 or 3 grammes of sand, placed in a paper thimble, such as is used with a Soxhlet, and covered with a layer of glass-wool or asbestos. The extracting vessel consists of a wide test-tube, through the cork of which passes a long vertical glass tube, somewhat narrowed at its lower extremity. Just above the contraction two lateral holes are made opposite one another, and a wire is pushed through the upper part of the thimble and through these holes, so that the indigo mixture is supported about halfway up the test-tube, and underneath the end of the glass tube which serves as condenser. Thirty or forty grammes of pure naphthalene are brought into the vessel, a metal jacket is put round it, and it is heated on a perforated asbestos plate for not more than three hours, till the drops of solvent fall from the thimble colourless. The apparatus is disconnected, the naphthalene melted, and a piece of copper wire with a coil at its end (to serve as a handle) is dropped in ; the mass is next solidified thoroughly, and loosened from the tube by a momentary insertion in boiling water. To separate the indigo-red and naphthalene from the indigotin, ether is used. The apparatus consists (A) of a round-bottomed flask with a side leading tube bent upwards ; (B) of a filter tube made of combustion tubing, with a bulb packed with asbestos above its narrow portion which descends through the cork of A ; (C) of a wide glass cylinder (rather wider than the test-tube previously employed) tapering to

a narrow tube which enters the top of B, and with a side-tube passing from its top downwards (externally) to meet the ascending tube from A. Above C is an inverted condenser. The mass of napthalene and indigotin is dropped into C, the test-tube is rinsed with 50 c.c. of ether, which, with another 150 c.c., is poured into A, and the apparatus is heated on the water-bath for about fifteen minutes till all the naphthalene and indigo-red are dissolved, and the indigotin itself remains on the asbestos filter. B is then dried at 100° C. and weighed; the bulb is next heated in a rapid current of air until the indigo has sublimed, and weighed again. The loss in weight represents the indigotin. The whole operation takes only five or six hours.

F. H. L.

The Solubility of Proteoses and Peptones in Alcohol. J. Effront. (*Bull. Soc. Chim.*, 1899, xxi., 676-680.)—Several methods of estimating the various products of the peptic digestion of albuminous substances have been based on their different solubility in alcohol of different degrees of strength, but the results obtained by one method do not agree with those given by another.

The author has made a number of experiments on this point, from which it appears that the degree of solubility of a series of peptonized products is largely dependent on the reaction of the solvent.

This point is illustrated in the following results obtained with the same crude peptone when treated with 95 per cent. alcohol acidified with different quantities of hydrochloric acid:

	Alcohol, 95%.	Peptone.	Acid.	Insoluble Part.		Ash in Insoluble Part.
		Grammes.	Centigrammes.	Grammes.	Per cent.	Per cent.
1 ...	100 c.c.	2	0	1·099	54·9	5·2
2 ...	100 c.c.	2	14	0·523	26·1	8·79
3 ...	100 c.c.	2	35	0·190	9·5	9·1

On increasing the quantity of acid a point is reached at which no precipitate whatever is obtained. On the other hand, it is possible to get results in close agreement if the alcohol employed be perfectly neutral. C. A. M.

The Estimation of Albumoses and Peptones. J. Effront. (*Bull. Soc. Chim.*, 1899, xxi., 680-683.)—The following method of analysis is based on the author's observations on the solubility of these substances in alcohol (see preceding abstract):

Fifty c.c. of a 5 per cent. solution of the crude peptone are neutralized with decinormal soda and allowed to stand for two hours. The precipitate of syntonin formed is collected on a weighed filter, washed with water and absolute alcohol, dried at 100° C., and weighed. The ash present is also determined and deducted.

A fresh portion of 50 c.c. of the original solution is neutralized in the same way, the liquid diluted to 55 c.c., and the syntonin filtered off after some time, but not washed. To 44 c.c. of the filtrate, corresponding to 40 c.c. of the original solution, are added 8 c.c. of normal hydrochloric acid, and then by degrees 250 c.c. of 95 per

cent. alcohol. After the addition of 8 c.c. of normal sodium hydroxide to the clear solution, the mixture is well shaken, and allowed to stand for two hours, and the resulting precipitate collected on a weighed filter, washed with 75 per cent. alcohol, dried at 100° C., and weighed as albumose.

The filtrate and washings are evaporated on the water-bath, and the residue, consisting of peptones and mineral matter, dried and weighed.

When the substance under examination contains a large proportion of mineral matter, it is advisable to determine the percentage of nitrogen in the different precipitates. In such cases it is also often possible to remove much of the mineral matter by allowing the acidified alcoholic solution to stand for some time before adding the alkali and filtering off the deposited salt.

In illustration of this method the author gives the following results of the analysis of a crude mixture of fibrin derivatives (practically free from ash) at various stages of continued peptonization :

	Original Mixture.	After Three Hours.	After Six Hours.	After Twelve Hours.	After Forty-eight Hours.
	Grammes.	Grammes.	Grammes.	Grammes.	Grammes.
Syntonin	1·14	0·30	0·25	0·2	0·0
Albumoses	1·82	1·869	1·780	1·452	1·41
Peptones	1·32	2·255	2·350	2·750	3·02

C. A. M.

The Estimation and Separation of Ferrocyanides and Carbonyl-ferrocyanides. J. A. Muller. (*Bull. Soc. Chim.*, 1899, xxi., 475-477.)—*A. Estimation of Ferrocyanides.*—The solution containing 4 to 5 grammes per litre is rendered slightly acid with nitric or acetic acid, and lead nitrate or lead acetate added so long as a precipitate results. After twelve hours the liquid is filtered and the precipitate washed with boiling water, dried, detached from the filter, and treated with a mixture of 5 parts of concentrated sulphuric acid and 3 parts of water. The crucible is covered and heated on a sand-bath to expel the excess of sulphuric acid, and then for some time in a muffle at a dull red heat. The residue of lead sulphate and ferric oxide is weighed, and the weight added to that obtained by treating the filter-paper in the same manner.

The combined results, multiplied by 0·5378, give the corresponding amount of potassium ferrocyanide.

In this way the author obtained 99·72 per cent. in nitric acid solution and 100·20 per cent. in an acetic acid solution of ferrocyanide.

B. Estimation of Carbonyl-Ferrocyanides.—An excess of pure cupric acetate is added to the dilute solution of the carbonyl-ferrocyanide, previously acidified with acetic acid. After a day the liquid is filtered and the precipitate thoroughly washed. It is then transferred to a platinum crucible and treated with dilute sulphuric acid (5 : 3). The crucible is heated at first gently on a sand-bath, and then more strongly, until the carbon is completely oxidized. The residue is dissolved in

fuming hydrochloric acid and the copper estimated as cupric sulphide by Rose's method.

The weight of cuprous sulphide, multiplied by 2·787, gives the corresponding amount of potassium carbonyl-ferrocyanide. In test experiments by this method the author obtained 99·12 per cent. of the theoretical amount.

Separation of Ferrocyanides and Carbonyl-Ferrocyanides.—It is not possible to estimate a ferrocyanide by simply precipitating it in the manner described above, when a carbonyl-ferrocyanide is also present in the solution, since some of the latter compound is simultaneously precipitated.

In such cases the author has found the following method satisfactory: The solution of the two ferrocyanides (4 to 5 grammes per litre) is slightly acidified with acetic acid (8 to 10 grammes per litre), and a solution of lead acetate added little by little, with constant shaking, until in slight excess. The vessel is left in a dark place for two or three days until the supernatant liquid is clear. This liquid is then decanted, filtered several times until bright, and an excess of cupric acetate added to precipitate the carbonyl-ferrocyanide.

The lead precipitate is dissolved in a slight excess of a solution of pure sodium hydroxide, and the solution poured into water containing sufficient acetic acid to leave the final liquid acid. The precipitate is left for about a day to settle, and twice redissolved and reprecipitated.

The final precipitate is then treated as described above in *A*; the filtrates are precipitated with cupric acetate, and the precipitates treated as in *B*.

In a test mixture of potassium ferrocyanide and carbonyl-ferrocyanide the author found by this method 99·37 per cent. of the former salt and 100·37 per cent. of the latter. C. A. M.

INORGANIC ANALYSIS.

Estimation of Arsenic in Paris Green. T. Smith. (*Journ. Amer. Chem. Soc.*, vol. xxi. [9], pp. 769-772.)—Two grammes of the substance are boiled for a few minutes with 100 c.c. of water and 2 grammes of sodium hydroxide, the solution being made up to 250 c.c. on cooling, and filtered. Fifty c.c. are then concentrated to one half, cooled to about 80° C., and treated with an equal volume of strong hydrochloric acid and 3 grammes of potassium iodide. After standing for about ten minutes, the precipitate is dissolved by an addition of water, and dilute sodium thiosulphate is added until the deep red colour of the solution just disappears. It is then neutralized with dry sodium carbonate, treated with an excess of sodium bicarbonate, and titrated with $\frac{N}{10}$ iodine, using starch solution as indicator.

To facilitate calculation, it is preferable to use an iodine solution 1 c.c. of which equals 0·005 gramme of arsenic trioxide, instead of decinormal iodine, the number of c.c. consumed, multiplied by 1·25, then giving the percentage of arsenic trioxide in the Paris green. Though the deep red colour developed during the titration helps to mask the end-point, the blue can be easily seen after a few trials, and duplicate tests agree to within 0·05 per cent. C. S.

Determination of Thallium as Chromate. P. E. Browning and G. P. Hutchins. (*Zeits. anorg. Chem.*, 1899, xxii., 380.)—This is an investigation of the process proposed by Crookes for precipitating thallium as thallous chromate by means of potassium bichromate in alkaline solution. The liquid should be heated to 70° or 80° C., made distinctly alkaline with potassium (rather than ammonium) carbonate, and then the bichromate added. After shaking well to make the precipitate cohere, the liquid is allowed to cool, filtered through asbestos in a Gooch crucible, and the chromate dried to constant weight over a small flame. Precipitation in the cold causes some of the chromate to pass through the filter, and although this can be partly avoided by adding ammonium nitrate, the hot treatment is preferable. The results are satisfactory.

By employing a known excess of a standardized bichromate solution, and estimating the portion of the reagent remaining in the filtrate from the thallous chromate, the process can be made volumetric. The filtrate is acidified with sulphuric acid and mixed with a measured excess of arsenious acid; when the colour has become bluish-green, the whole is rendered alkaline with potassium bicarbonate, and the unaltered arsenious acid titrated with iodine. The original bichromate is standardized similarly. Allowing for the high molecular weight of thallous oxide in comparison with chromic acid, this method is also fairly satisfactory. F. H. L.

Electrolytic Determination of Zinc in Presence of Manganese. E. J. Riederer. (*Journ. Amer. Chem. Soc.*, vol. xxi. [9], pp. 789-792.)—The author employed the following reagents: Lactic acid solution containing 0·937 gramme per 1 c.c.; ammonium lactate solution, 0·5 gramme per 1 c.c.; and crystalline ammonium sulphate. The total volume of solution was 230 c.c.; the silvered platinum cathode surface, 150 sq. cm.; distance between anode and cathode, 1·5 cm.; temperature, 26° C.; strength of current, 4 volts, equal to 1·5 volts and 0·20 to 0·26 ampère for each 100 sq. cm. of cathode area; time, four to five and a half hours. Continued stirring gives a more rapid and uniform deposit of zinc. No manganese was deposited on the cathode, though in several cases small amounts of the hydroxide were found on the anode.

The zinc deposit is washed with water, alcohol, and finally with pure ether, and weighed after drying in a hot-water oven and desiccator—this part of the operation taking only *five minutes*. The liquid assumed a dark colour during electrolysis when manganese was present, but this disappeared shortly after the stoppage of the current.

The results obtained with zinc sulphate and manganese sulphate varied by not more that 0·10 per cent. of the theoretical value of the zinc. C. S.

New Volumetric Method for Magnesium. R. K. Meade. (*Journ. Amer. Chem. Soc.*, vol. xxi. [9], pp. 746-752.)—The solution, free from any large excess of ammonium chloride or ammonium oxalate, is strongly agitated in a flask with

one-third its volume of strong ammonia and 50 c.c. of sodium arsenate for about ten minutes. The precipitate is washed with dilute ammonia (1 : 3) until the washings just cease to react for arsenic, and then dissolved in dilute hydrochloric acid (1 : 1), the filter being washed with the same until a total volume of 75 or 100 c.c. is obtained. When cool, the solution is treated with 3 to 5 grammes of potassium iodide (free from iodate), and titrated with standard sodium thiosulphate until the iodine fades to pale straw colour, whereupon starch is added and the titration continued until the colour of the iodide of starch is discharged. If preferred, an excess of thiosulphate may be added, and titrated back with starch and standard iodine until the blue colour is developed. The brown precipitate formed when potassium iodide is added to the acid solution redissolves in presence of thiosulphate. C. S.

Estimation of Sulphuric Acid in Presence of Iron. F. W. Küster and A. Thiel. (*Zeits. anorg. Chem.*, 1900, xxii., 424.)—This is the third contribution, partly polemical, published by the present authors, dealing with the question of avoiding contamination of barium sulphate with iron when sulphuric acid is precipitated by means of barium chloride from a solution containing that metal. The authors sum up their results and the general position as follows: If sulphuric acid is precipitated from a hot liquid which contains ferric ions, the barium sulphate is mixed with the compound $BaFe_2(SO_4)_4$. The formation of this double salt may be avoided either by removing the ferric ions or by producing conditions in which the said double salt is not formed. The ferric ions can be removed either by precipitation with ammonia—filtration being unnecessary—or they can be converted into some complex form—*e.g.*, by the addition of ammonium oxalate—in which they are no longer harmful. The conditions that prevent the formation of the double salt are either precipitation in the cold or a reversed precipitation—that is to say, in hot liquids the addition of the mixture of ferric salt and sulphuric acid to the solution of barium chloride. Conversion of the ferric ions into ferrous ions by treatment with zinc, etc., is not a perfect cure for the trouble, since, although the main source of error is thus avoided, the barium sulphate may still be contaminated with zinc sulphate or ferrous sulphate. (*Cf.* ANALYST, xxiv., 137 and 164). F. H. L.

Iodometric Analysis of Mixtures of Chlorates and Hypochlorites. H. Ditz and H. Knöpfelmacher. (*Zeits. angew. Chem.*, 1899, 1195 and 1217.)—In carrying out the process indicated by the above title, it is customary to decompose the chlorates by heating them with hydrochloric acid and potassium iodide in a closed vessel. The authors find that the reaction, which should be represented by the equation,

$$KClO_3 + 6HCl + 6KI = 6I + 7KCl + 3H_2O,$$

does not proceed quantitatively at ordinary temperatures, nor can it be made to do so by varying the strength of the acid or the time of treatment. If, however, the potassium iodide is replaced by bromide, and if every precaution is taken to avoid loss of bromine vapours, the process becomes quite accurate. A 1-litre bottle is

provided with a hollow glass stopper, which carries centrally a pear-shaped separating funnel (with its own stopper and stopcock), and laterally a small washing flask, the exit tube of which also has a stopper. The main bottle is also fitted with a plain stopper for use when the rest of the apparatus is not being employed; and thus everything is composed of glass. The washing flask is charged with potassium iodide solution. In the bottle are placed the chlorate (0·06 to 0·08 gramme of $KClO_3$) and 20 c.c. of a 5 per cent. solution of potassium bromide; 50 c.c. of strong HCl are run in through the funnel, and the exit stopper of the washing flask is quickly inserted. After an hour, 200 or 300 c.c. of water are added, 20 c.c. of 5 per cent. potassium iodide are introduced, the mixture is well shaken, the iodide in the side flask is blown back into the rest of the liquid, and the whole is titrated with decinormal thiosulphate.

The process is equally available for pure chlorates or for chlorates mixed with hypochlorites, the amount of the latter being ascertained separately. If, however, the proportion of chlorates is very small in comparison with the hypochlorites, as, for example, in bleaching powder, the latter should be destroyed first. The authors doubt whether any of the methods hitherto suggested for this purpose are free from objection; perhaps treatment with carbon dioxide, hydrogen peroxide, or barium peroxide is the best. This matter is being further investigated, and will be dealt with later. F. H. L.

Estimation of Nitrogen in Nitrates. M. Schmoeger. (*Chem. Zeit.*, 1899, xxiii., 829.)—The author has investigated the accuracy of four processes for the estimation of nitrogen by employing them on specimens of potassium and sodium nitrate of known purity. The methods examined were: I., the Kühn or Raab process (reduction with zinc and iron in alkaline solution); II., the Ulsch process; III., the Devarda process (reduction in alkaline solution with an alloy of aluminium, copper, and zinc); and, IV., the Kjeldahl-Förster process. His results, as shown by the table, prove Devarda's plan to be the best, Kühn's to be the worst. Blank analyses are required in all cases, and in II. the purity of the iron must be ascertained (*cf.* ANALYST, 1899, xxiv., 80).

	$NaNO_3$	KNO_3
I.	16·24	13·63
II.	16·37	13·80
III.	16·41	13·83
IV.	16·41	13·80
Theory	16·50	13·88

F. H. L.

A New Reagent for Detecting and Estimating Nitrites in Water. H. Erdmann. (*Zeits. angew. Chem.*, 1900, 33.)—This substance consists of amidonaphtholdisulphonic acid [$NH_2 : OH : HSO_3 : HSO_3 = 1 : 8 : 4 : 6$], which is prepared by nitrating, reducing, and heating with sodium hydroxide the naphthalene (1 : 3 : 5) trisulphonic acid lately described by the author (*Berichte*, 1899, xxxii., 3186). Fifty c.c. of the water are mixed with 5 c.c. of a 0·2 solution of sodium aniline-*p*-sulphonate containing hydrochloric acid, and after ten minutes (diazotation is not completed instan-

taneously in such very weak liquids) 0·5 gramme of solid amidonaphtholdisulphonic acid is added in the form of its acid alkali-metal salt. In presence of nitrous acid a brilliant Bordeaux-red colour appears, which attains its maximum intensity in one hour. The test may be rendered quantitative by colorimetric comparison with $\frac{N}{1,000,000}$, $\frac{N}{100,000}$, and $\frac{N}{10,000}$ solutions of sodium nitrite; or by means of a coloured paper scale previously prepared. These weak nitrite solutions should be diluted from the normal liquid when they are required.

As regards the determination of nitrites when nitrates are present, Gries's test with *a*-naphthylamine, which is officially recognised in Alsace and Lorraine, is not suitable for the examination of water: the orange colouring matter is very insoluble, and is often thrown down in hard or saline water as a flocculent precipitate, which renders colorimetry impossible. Riegler's process (ANALYST, 1897, xxii., 305) is not recommended by Erdmann: naphthionic acid and numerous easily diazotizable substances give fluorescent liquids, and the fluorescence is inconvenient. The above naphthol compound does not fluoresce under the special conditions of the test; moreover, oxidizing agents such as ferric chloride colour it yellow, a tint that cannot be mistaken for the red caused by nitrous acid. Phenylenediamine and analogous bodies yield brown colours both with nitrous acid and with oxidizing agents; and for similar reasons the starch-zinc iodide test, still popular in many bacteriological laboratories, must be rejected.

Free samples of Erdmann's new reagent ready for use can be obtained from J. F. Schwarzlose Söhne, Markgrafenstrasse, 29, Berlin, S.W., and the author appeals to analysts to test its value. F. H. L.

Notes on the Determination of Sulphur in Pig Iron. M. J. Moore. (*Journ. Amer. Chem. Soc.*, vol. xxi. [11], pp. 972-975.)—The author finds that the physical treatment of the sample is largely responsible for the inaccuracies experienced in the volumetric determination of the sulphur, "shot samples" taken from the cupola (because of the difficulty of drilling the cast metal) giving an apparently lower sulphur content than those caught in a sand mould from the mixer. On comparing samples taken by the two methods from the same charge in the mixer, the results of the volumetric tests averaged 0·018 higher for the sand-mould samples, thus showing the unsuitability of "shot" sampling for this mode of analysis. In the gravimetric method, on the other hand, the results were practically identical in both cases. With Bessemer iron low in sulphur the difference (average 0·005 in "sand" samples) between the gravimetric and volumetric results is not so apparent until No. 2 grade is reached. C. S.

Preservation of Hübl's Reagent. R. Bolling. (*Amer. Chem. Journ.*, vol. xxii. [3], pp. 213, 214.)—A standard solution of the reagent was divided and stored under different conditions, viz.:

A1.—In clear glass bottles one-third to a quarter full, loosely stoppered, frequently opened, and exposed to daylight.

A2.—In similar bottles, quite full, closely stoppered, and exposed to daylight.

A3.—In amber-coloured bottles, filled, closely stoppered, and kept in darkness.

A similar solution, but prepared with absolute alcohol, was also stored in filled, closely-stoppered bottles, exposed to daylight (B2) and in darkness (B3).

On testing 5 c.c. with sodium thiosulphate, the initial quantity of free iodine (0·1250 gramme) was found to progressively decrease with storage, the amounts remaining at the end of eight weeks being: A1, 0·0641; A2, 0·0629; A3, 0·0702; B2, 0·0811; B3, 0·0835 gramme, the conditions having but little influence on the result. C. S.

Perezol: a New Indicator. M. Duyk. (*Ann. de Chim. Anal.*, 1899, iv., 372-374.)—Perezol, or pipitzahoic acid, is a compound obtained from the rhizome of the Mexican plant *perezia adnata*. It is an extremely sensitive indicator, for even the weakest alkalies yield with it salts of a violet to black hue. When distilled water containing a drop of the indicator (0·5 per cent. alcoholic solution) is boiled in a glass vessel, the liquid becomes rose-coloured owing to the traces of alkali dissolved. In addition to the fixed and volatile alkalies, the colour of perezol is also changed by alkaloids.

The alkaline, rose-coloured liquid becomes colourless, without any transition stage on treatment with mineral acids, carbonic acid, and organic mono- and poly-basic acids. Boric acid, however, is an exception, and an addition of glycerin is required for the titration. Mineral salts of the strong acids do not affect perezol, but carbonates, bicarbonates, borates, and acetates produce a rose or mauve tint. Ammoniacal salts are also without influence. C. A. M.

APPARATUS.

A Simple Method of Protecting Platinum Wires fused into Glass Tubes. A. Rosenheim. (*Berichte*, 1899, xxxii., 2570, 2571.)—The device adopted by the author to prevent platinum wire fused into glass from being broken is shown in the accompanying figures.

The platinum wire *D*, fused into the vessel *A*, is surrounded on the exterior by a glass tube, *R*, fused on to *A*. This tube is partially filled with mercury, *Q*, in which is immersed the connecting wire, *L*, of the battery. In Fig. 2 and Fig. 3 the tube *R* is adapted for the protection of a wire fused into the side or bottom of a vessel. The tube (Fig. 3) can be easily filled with mercury with the aid of an air-pump. In order to prevent the mercury from being spilled, the opening of the tube may be narrowed, as shown in Fig. 2. C. A. M.

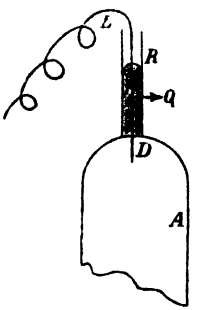


Fig. 1

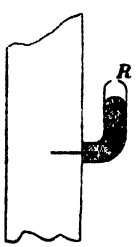


Fig. 2.



Fig. 3.

APPEAL ON ACTION SUPPOSED TO HAVE BEEN TAKEN UNDER WRONG SECTION OF THE FOOD AND DRUGS ACT.

QUEEN'S BENCH DIVISION.

(Before Mr. JUSTICE CHANNELL and Mr. JUSTICE BUCKNILL.)

BEARDSLEY *v.* WALTON AND CO.

(*From the* TIMES *of February* 9, 1900.)

THIS was an appeal by case stated from Justices of Wiltshire. An information was laid by the appellant against the respondents charging them with selling to the prejudice of the purchaser a drug—to wit, camphorated oil—which was not of the nature, substance, and quality demanded by the purchaser, the said camphorated oil only containing 8 parts per cent. by weight of camphor, or less than half the quantity of camphor proper to camphorated oil as described in the British Pharmacopœia, contrary to section 6 of the Sale of Food and Drugs Act, 1875. The Justices dismissed the information on the ground that the camphorated oil was a compounded drug, and that consequently the summons could only be issued under section 7 of the Act. Section 6 of the Act provides as follows: "No person shall sell to the prejudice of the purchaser any article of food or any drug which is not of the nature, substance, and quality of the article demanded by such purchaser under a penalty not exceeding £20, provided that the offence shall not be deemed to be committed under this section in the following cases—that is to say . . . (3) Where the food or drug is compounded as in this Act mentioned." Section 7 is as follows: "No person shall sell any compound article of food or compounded drug which is not composed of ingredients in accordance with the demand of the purchaser under a penalty not exceeding £20."

Mr. F. R. RADCLIFFE, for the appellant, contended that camphorated oil was not a compounded drug, because the oil was only a vehicle to enable the camphor to be applied. He also contended that subsection 3 of section 6 did not prevent section 6 applying in the case of compounded drugs sold to the prejudice of the purchaser. He cited White *v.* Bywater (19 Q.B.D., 582) and Houghton *v.* Taplin (13 The *Times* Law Reports, 386).

Mr. BRUCE WILLIAMSON, for the respondent, contended that the effect of subsection 3 was to take offences in regard to compounded drugs out of section 6 and to bring them under section 7.

The Court on Wednesday allowed the appeal.

Mr. Justice CHANNELL said that the difficulty of the case arose from the use by the Justices of the word "consequently." If they had said that camphorated oil was a drug compounded as in this Act mentioned, then the consequence might have followed. The only possible explanation of the words "as in this Act mentioned" was that there was in the Bill a clause dealing with compounded drugs which was afterwards struck out. Their Lordships were not entitled to know that that was the case, but they were entitled to assume so from the language as it stood. The only section in the Act which it could be read as referring to was section 7, which did not in language correspond with it, but did deal with compounded drugs. If so, it must mean that nothing should be an offence under section 6 with reference to compounded drugs unless it was also an offence under section 7. Whatever meaning subsection 3 bore, this offence was equally within section 6.

Mr. Justice BUCKNILL concurred.

THE ANALYST.

APRIL, 1900.

PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS.

THE monthly meeting of the Society was held on Wednesday evening, March 7, in the Chemical Society's Rooms, Burlington House. The President, Mr. W. W. Fisher, M.A., occupied the chair.

The minutes of the previous meeting were read and confirmed.

Certificates of proposal for election to membership in favour of Messrs. J. E. Kirkpatrick, A. J. Murphy and A. A. Ramsay were read for the second time; and certificates in favour of Messrs. Thomas Eustace Hill, M.B., B.Sc. (Edin.), F.I.C., Shire Hall, Durham, Medical Officer of Health for the County of Durham; Thomas Macara, 6, West Bank, Hillhead, Glasgow, assistant to Dr. John Clark; and William E. F. Powney, 67, Barrett's Grove, Stoke Newington, chief assistant to Mr. G. T. Holloway, were read for the first time.

Messrs. Norman Parr Booth, John Macallan and C. T. Tyrer were elected members of the Society.

The following papers were read: "The Determination of Carbon and Sulphur in Steel," by Bertram Blount; "Maize Oil," by Rowland Williams; and a "Note on the Assay of Creosote," by A. D. Hall, M.A.

THE ANALYSIS OF GOLDEN SYRUP.

By NORMAN LEONARD, B.Sc.

IN the December number of the *Journal of the Society of Chemical Industry* (1899, xviii., p. 1091) there appears, under the title of "Notes on the Analysis of Some Sugar Syrups," a criticism, by Messrs. A. K. Miller, Ph.D., and J. H. Potts, of a recent paper on the analysis of golden syrup by Mr. R. Bodmer, Mr. H. M. Smith, and myself (ANALYST, 1899, xxiv., p. 253). Whilst admitting the fairness, from their own standpoint, of the objections raised to our work by Dr. Miller and his colleague, I wish to emphasize the fact that our paper was communicated to the Society of Public Analysts, and was written from the point of view of the Public Analyst; it was not in any way intended as an introduction of new processes for the exact analysis of sugar syrups. At the time we wrote, the analysis of golden syrup was new to Public Analysts, *qua* Public Analysts, and since numerous inquiries addressed

to one of us indicated a lack of information on the subject, we thought it useful to outline a scheme of analysis, the details of which might be altered or extended to suit individual preferences, and to show how, by a balancing of probabilities, a fairly safe and generally intelligible opinion as to the nature of a sample might be arrived at. It is doubtful if any Public Analyst would care to certify the amount of glucose added to golden syrup more accurately than to the nearest 5 per cent., and even then, seeing the variable composition of glucose and golden syrups, the adoption of such expressions as "*about* 50 per cent. of glucose syrup" or "*at least* 70 per cent. of glucose syrup" is advisable. It will thus be seen that the meanings attached to the word "approximate" by Messrs. Miller and Potts on the one hand, and by ourselves on the other, are rather different, and that consequently many of the objections raised to our analyses lose somewhat of their force.

Messrs. Miller and Potts seem not to appreciate the position of the Public Analyst, who is bound by the form of certificate prescribed by the Sale of Food and Drugs Acts to give a quantitative expression of the composition of an article which he believes to be adulterated; he is not permitted to "be content with a qualitative test." This being so, recourse must be had to the method of averages, so much deprecated by Dr. Miller and Mr. Potts, which those whose business it is to examine products of variable composition are often obliged to employ. An exact determination of all the constituents of a sugar syrup would doubtless be of great interest to the chemist and to the manufacturer, but such an analysis would, as a rule, be quite unintelligible both to the magistrate and to the vendor charged with adulteration. The analytical results would therefore require to be expressed in terms of such comparatively familiar substances as cane-sugar syrup and glucose syrup, and this would again necessitate the use of averages, for it would be impossible to say accurately how the water, ash, and organic matter were distributed between the two syrups in a mixture.

Respecting the errors introduced into the estimation of the water by neglecting the influence of the ash on the specific gravity of the syrup solution, I may observe that the amount of water present is, to the Public Analyst, of very little importance. We were aware of this source of error, but it may be pointed out that large amounts of ash are, I believe, rarely found except in crude, genuine syrups which would be passed without comment by a Public Analyst, whilst in those samples which are largely adulterated with glucose syrup the ash is usually low, and the resulting error, which here becomes of more importance, is also small, and comparable with that inherent in the divisor process when applied to such complex mixtures as those in question.

Although not claiming so much accuracy for our analyses as Dr. Miller appears to have thought was the case, it may be of interest to make some remarks on the minus values obtained for invert sugar in No. 4 sample and some others. These values, which we had understood were not uncommonly obtained in the indirect analysis of complex and impure syrups, had not escaped our notice, and we had followed the matter up even farther than our critics seem to have done. For although it is quite true, taking $i = 0$ in the equation $K = i + 0{\cdot}53x$, that the *maximum* percentage of glucose (on the basis of the 53 factor) in No. 4 sample is 62·6, it is

possible to look at the matter from another point of view. Thus, in the equation $[\alpha]_D = \frac{66{\cdot}5s + 134x - 23{\cdot}1i}{100}$, if there is no invert sugar i must equal 0 and x (glucose) becomes 75·8; and this is the *minimum* percentage of glucose on the basis of the 134° factor. This sample is not improbably adulterated with a glucose syrup having a lower reducing power than the average assumed by us, and we have here an example of the discrepancies inseparable from the use of averages, which, however, as already stated, must necessarily be employed.

It should be remembered that the Public Analyst, whilst making use of average analyses as a basis for calculation and comparison, is, or should be, fully aware of the greater or less amount of reliance to be placed on them, and is accustomed to regard his results from various points of view, knowing that the most elaborate and accurate analysis may lead him astray unless due regard is paid to modes of interpretation and to other circumstances than analytical details. The statements of the proximate composition of the mixed syrups given at the close of our paper (ANALYST, xxiv., p. 257) are to be regarded in this light, and, although more accurate methods are greatly to be desired, Messrs. Miller and Potts appear unable to suggest a process better adapted to the wants of the Public Analyst than that described by Mr. Bodmer, Mr. Smith, and myself.

TREACLE, OR GOLDEN SYRUP.

By E. W. T. JONES.

AT the request of certain friends, I beg to lay before the Society of Public Analysts my method of treating the subject of the adulteration of golden syrup with starch glucose syrup, because, although there have been several excellent papers bearing on the subject, I believe my view of the question simplifies matters without detracting from the practical accuracy obtainable by more detailed methods. Of course, I do not advocate blindly applying my formula to every sample that may be presented. I am taking things as we find them at present, and a public analyst will naturally adapt himself with intelligence to any varying conditions which may arise.

Golden syrup, whether the natural by-product of the sugar industry or the artificial product from refined sugar, consists essentially of sucrose and invert sugar, the difference between the two classes of products consisting mainly in the relative proportions of sucrose and invert sugar. By converting the sucrose into invert sugar considerable uniformity, from a polarimetric point of view, is obtained in the case of genuine samples, which at the same time are made to exhibit a greater divergency from glucose syrup, which always has a high dextro-rotation, genuine samples of golden syrup being, after inversion, lævorotatory. The following two samples will illustrate this statement. The one is Crosfield's genuine cane syrup, and the other is Lyle's golden syrup, the former containing nearly 40 per cent. of sucrose and the latter about 30 per cent.:

	Specific Rotatory Power, at 17·5° C.			
	Original.		After Inversion.	
	$[\alpha]_J$	$[\alpha]_D$	$[\alpha]_J$	$[\alpha]_D$
No. 1. Crosfield's	+26·90	+24·21	−12·30	−11·07
No. 2. Lyle's	+16·13	+14·52	−15·37	−13·83

Glucose syrup is not sensibly altered by a careful inversion treatment. The sample in my possession, which is representative of what is used, has a specific rotatory power at 17·5° C. of $[\alpha]_J + 121$ or $[\alpha]_D + 109$. I therefore suggest the following simple formula for arriving at the percentage of glucose syrup in a sample of golden syrup, if the same *after inversion* has a *dextro*-rotation:

$$[\alpha]_J \frac{([\alpha]_J \textit{ after} \text{ inversion} + 12{\cdot}3)100}{133{\cdot}3} = \text{per cent. glucose syrup.}$$

$$[\alpha]_D \frac{([\alpha]_D \textit{ after} \text{ inversion} + 11)100}{120} = \text{per cent. glucose syrup.}$$

A sample of golden syrup was sent to me, said by the manufacturer to contain 18 per cent. glucose syrup. It had a specific rotatory power *after* inversion of:

$[\alpha]_J$ +12·3 $[\alpha]_D$ +11·07

By the above formulæ, it shows 18·4 per cent. of glucose syrup.

I have taken the figure of the No. 1 syrup because these darker-coloured syrups are those used generally to mix with glucose syrup.

The way I actually proceed with a sample is to make a 10 per cent. solution at 15·5° C., and take the specific gravity. This should not be sensibly less than 1032, which indicates 20 per cent. of water, for I have found by carefully estimating the water in vacuo, or by drying after incorporation with thirty times its weight of sand, that the divisor 4 for specific gravity over 1,000 in a 10 per cent. solution is practically correct for the solid matter. The divisor 3·86 is correct for about 10 per cent. solution of *pure sucrose*, but gives low results, to the extent of about 3 per cent. of water in the case of ordinary golden syrup, thus all Messrs. Bodmer, Leonard and Smith's estimations of water given in their paper (Analyst, vol. xxiv., p. 255) are about this much too low.

The 10 per cent. solution above mentioned (if I wish to estimate the sucrose) I raise to a temperature of 17·5° C., and examine in the polarimeter in 100-millimetre tube. Fifty c.c. of the same solution + 5 c.c. N acid are heated in a 100 c.c. flask in boiling water for twenty minutes, then cooled, and 5 c.c. N soda are added, and the bulk made up to 100 c.c. at 15·5° C. This solution is examined through the 100-millimetre tube at the proper temperature, 17·5° C., and the reading multiplied by two to make it correspond to the 10 per cent. solution.

I do not use the 200-millimetre tube, since the inverted solution is often too dark, and I purposely avoid the use of decolorizing agents. Mine is a Ventzke-Scheibler polarimeter, and, as a matter of fact, I generally for this purpose work with scale degrees.

The sample referred to as adulterated with 18 per cent. glucose syrup, gave for the 10 per cent. solution after inversion +3·2 scale divisions:

$$\frac{\text{Plus scale divisions observed in sample after inversion} + 3{\cdot}2}{0{\cdot}347} = \text{per cent. glucose syrup.}$$

Thus it comes to 18·4 per cent.

For the copper reduction, if it is required, I take 10 c.c. of the first solution and make up to 100 c.c.; 20 c.c. of this (=0·2 gramme of the sample) I add to 50 c.c. of Fehling solution diluted with 30 c.c. of water, which has acquired the temperature of, and still stands in, *boiling* water. The mixture is allowed to remain twelve minutes, and then filtered through an ashless filter, well washed, dried, ignited in a porcelain crucible (very slowly at first, only *just smouldering* the filter away. The CuO then does not cohere, and is fully oxidized). The CuO × 0·4307 (Log $\bar{1}$·63414) = invert sugar. Of the inverted solution I take 20 c.c. and make up to 100 c.c., and take 20 c.c. (=0·2 gramme of the sample), add to Fehling solution, and proceed as above. The quantity of CuO found in the preceding experiment is deducted from that obtained after inversion, and the difference × 0·4091 (Log $\bar{1}$·61186) = cane sugar in 0·2 gramme of the sample.

I have examined a great many samples very thoroughly, and find that the simple method I gave at the commencement of this paper has afforded very consistent results. It has been suggested that the alcohol test for dextrin is of no use, but if it is always applied in a definite way, viz., taking 2 c.c. of the 10 per cent. solution and adding 20 c.c. of strong (miscible) methylated spirit, the *white* cloudiness formed is quite distinguishable from the occasional turbidity of some samples not containing dextrin.

ANALYSIS OF A SAMPLE OF TREACLE AND OF SO-CALLED GOLDEN SYRUP.

By Charles George Matthews, F.I.C., and A. Hyde Parker.

(*Read at the Meeting, February* 7, 1900.)

Values used in Calculating the two Analyses.

The usual divisor for carbohydrates, 3·86.

Value of a 1 per cent. solution of the sugar observed in a 200-millimetre tube:

	Soleil-Ventzke-Scheibler Divisions.		Laurent Degrees.
Maltose	8·02		2·76
Dextrin	11·56		3·98
Dextrose	3·05	× 0·344 =	1·05
Gallisin	4·85		1·67
Cane sugar	3·84		1·32

Cupric-reducing values:

	CuO equivalent to 1 Gramme of Substance.
Maltose	1·37 grammes
Dextrose	2·46 ,,
Gallisin	1·01 ,,

SAMPLE OF TREACLE (FULL COLOUR, GOOD FLAVOUR, BUT SLIGHTLY SALINE).

A. *Calculation of the Gravity due to Ash.*—Five grammes of the sample yielded 0·373 gramme of non-sulphated ash, equal to 7·46 per cent. This was shaken up with 100 c.c. of water and the specific gravity of the mixture found to be 1,003·25; by division $\frac{3{\cdot}25}{0{\cdot}373} = 8{\cdot}7$ was obtained as solution factor.

Another 5 grammes yielded 0·4485 gramme of sulphated ash, equal to 8·97 per cent., and this, similarly treated, gave a mixture with a specific gravity of 1,004·35 and a solution factor of 9·7.

The mean of these two percentages of ash was 8·21, and the mean solution factor 9·2.

Determination of the Dry Solids.—The specific gravity of a 10 per cent. solution of the sample was 1,032·10, and the mean specific gravity due to ash in a solution of the same strength was 1,007·55, therefore the specific gravity due to carbohydrates was 1,024·55, which, divided by 3·86, gives 63·6 per cent. of carbohydrates, and this figure added to that of the ash gives 71·81 per cent. total solids.*

B. *Determination of the Cane Sugar.*—One gramme of pressed yeast was added to 50 c.c. of a 10 per cent. solution of the sample, the mixture kept at a temperature of 52° C. for five hours, boiled to destroy bi-rotation, made up to bulk, filtered, and examined in a 200-millimetre tube.

	Soleil-Ventzke-Scheibler Divisions.	Laurent Degrees.
Before inversion	... +16·16	+5·56
After ,, ...	... − 6·33	−2·18
Difference ...	... +22·49	+7·74

The Soleil-Ventzke-Scheibler divisions, 22·49 × 10 and divided by 5·02,† or the Laurent degrees, 7·74 divided by 0·1727, give 44·8 per cent. of cane sugar.

C. *Cupric-reducing Power.* — Five grammes of the sample were dissolved in 100 c.c. of water, and the amount of cupric reduction in 2 c.c. estimated gravimetrically by the filter-paper method and weighing as cupric oxide.‡ The weight found 0·0305 grammes × 100 gives 3·05 for a 10 per cent. solution. This figure multiplied by 10 and divided by 2·36§ = 12·92 per cent. reducing sugars.

* Heron (*Trans. Fed. Insts. Brewing*, 1896, p. 449) regards the sulphated ash as accurately representing the ash without any correction, and he takes the solution factor for the ash as being about double that for the carbohydrates (approximately eight), but seeing that the sulphated ash is perceptibly higher than the plain ash, and the solution factor also higher, the number eight would in our view be sufficiently accurate as applied to the sulphated ash.

† Divisor for 1 per cent. cane sugar solution in 200-millimetre tube when converted into invert sugar.

‡ *Vide* Morris, *Trans. Fed. Insts. Brewing*, 1898, p. 164.

§ Mean cupric-reducing power per 1 gramme of invert sugar. (Levulose = 2·26.)

D. *Estimation of the Matter disappearing during Fermentation.*—A few grammes of pressed yeast were added to 150 c.c. of a 10 per cent. solution of the sample, and kept at about 18° C. until fermentation ceased (about seventy-two hours). The solution was then found to have the following constants: Optical activity, 0·0; cupric-reducing power (K_{100}) (10 per cent. solution), 0·65.

The specific gravity lost during fermentation was:

Original specific gravity	1,032·10
Extract gravity after fermentation	1,010·78
	21·32

This figure, $21{\cdot}32 \times 10 \div 3{\cdot}86 = 55{\cdot}2$ per cent. sugar which has disappeared during fermentation.

This figure, 55·2, may be compared with that obtained by adding the cane sugar and reducing sugar estimation, viz.:

Cane sugar	44·0
Reducing sugar	10·17
	54·97

The residual cupric-reducing power left after fermentation may be regarded as due to unfermented dextrose and lævulose, and the mean reducing value of these two reducing sugars is 2·36 (1 gramme invert sugar = 2·36 grammes copper oxide). Consequently $0{\cdot}65 \times 10 \div 2{\cdot}36 = 2{\cdot}75$ per cent. of a mixture of the two sugars, having an optical activity of 0·0. This calculated out to a mixture of 1·78 per cent. dextrose and 0·97 per cent. lævulose.*

The composition of the fermented matter consisted therefore of:—

Reducing sugars calculated from original cupric-reducing power	12·92	per cent.
,, ,, left unfermented	2·75	,,
,, ,, disappeared during fermentation	10·17	,,

Estimation of the Reducing Sugars removed by Fermentation.—The amount of these is found by subtracting the polarimetric reading corresponding to the cane sugar from that of the original 10 per cent. solution of the sample:

	Soleil-Ventzke-Scheibler Divisions.	Laurent Degrees.
Original solution in 200-millimetre tube	16·16	5·56
Deviation due to cane sugar ($4{\cdot}48 \times 3{\cdot}84$) ...	17·20	5·92
Difference	−1·04	−0·36

The reducing sugars were found previously to have a copper-reducing value of 2·4 in a 10 per cent. solution, and, since their optical activity was −1·04, they consisted of a mixture of 4·83 parts lævulose and 5·34 dextrose. These amounts added to the same sugars which were left unfermented equal 5·80 per cent. lævulose and 7·12 per cent. dextrose. (For mode of calculation, see previous footnote.)

* The composition of the mixed sugars is calculated as follows: Let x = the dextrose and $(2{\cdot}75 - x)$ = the lævulose, then $x \times 52{\cdot}8 + (2{\cdot}75 - x) \times -95{\cdot}65 = 0$, and $x = 1{\cdot}78$.

The complete analysis was as follows :

Cane sugar	44·80
Lævulose	5·80
Dextrose	7·12
Ash	8·21
Unaccounted for = inactive matter	5·87
	71·80
Difference = moisture	28·20
	100·00

Had the results been calculated in the old way, viz., by dividing the weight about 1,000 of the 10 per cent. solution by 3·86, we should have the following :

$$\text{Specific gravity } 1{,}032{\cdot}1 \text{ and } \frac{32{\cdot}1}{3{\cdot}86} \times 10 = 83{\cdot}16 \text{ per cent.};$$

83·16 would then have been formerly taken as the true solids, and the difference as moisture, and the above analysis (excluding any question of newer methods and values used in calculating the cane sugar, dextrose, and lævulose) would have become :

SUMMARY.

Cane sugar	44·80
Lævulose	5·80
Dextrose	7·12
Ash	8·21
Inactive matter	17·23
Difference = moisture	16·84
	100·00

SAMPLE OF SO-CALLED GOLDEN SYRUP (FULL GOLDEN COLOUR, PLEASANT FLAVOUR).

A. Five grammes of the sample yielded 0·067 grammes of sulphated ash = 1·34 per cent.

Specific gravity corresponding to ash in 10 per cent. solution 0·134 × 8 = 1,001·07.

Specific gravity of 10 per cent. solution of sample	1,030·40
Correction for ash	1·07
	1,029·33
29·33 ÷ 3·86 × 10 = Carbohydrates per cent.	75·92
Ash	1·34
Total solids	77·26

B. By inversion with yeast, as in the former case, 11·3 per cent. of cane sugar was found.

	Soleil-Ventzke-Scheibler Divisions.	Laurent Degrees.
Deviation in 200-millimetre tube for 10 per cent. solution of sample	+53·6	+18·4

C. Cupric-reducing power of 100 c.c. of 10 per cent. solution, 7·65 grammes CuO.

D. Specific gravity of 10 per cent. solution before fermentation ... 1,030·4
Extract ,, ,, ,, after ,, ... 1,013·5

Loss ... ... 16·9

16·9 ÷ 3·86 × 10 = 43·70 per cent. fermented.
33·56 ,, unfermented.

77·26 ,, total solids.

	Soleil-Ventzke-Scheibler Divisions.	Laurent Degrees.
Deviation in 200-millimetre tube after fermentation	+31·2	+10·7
Cupric reduction in 100 c.c.	1·544 grammes	

E. *Determination of combined Dextrin* (*Amyloïn-dextrin*).—Any maltose existing in combination as malto-dextrin would reveal its presence by an increased reducing power after treatment of the fermented solution with cold-water malt-extract,* and this released maltose would be removed by a subsequent fermentation with yeast. To 100 c.c. of the fermented 10 per cent. solution 10 c.c. of malt-extract were added, and the mixture kept at a temperature of 55° C. for two hours.

A considerable increase in the cupric-reducing power ensued (= 18·3 per cent. of dextrin), but inasmuch as the solution after treatment with malt extract was not susceptible of fermentation to an appreciably further point than the non-treated portion, we do not consider ourselves justified in using the figure so obtained for dextrin.

The same rather curious fact has been noticed in the case of two or three other syrups which had been undoubtedly manufactured from cereals or cereal starch.

This consideration of the failure of the solution treated with malt-extract to ferment further than the untreated solution, also affects the question of the combined maltose, which should be—like the maltose produced from hydrolysed dextrin—rendered fermentable by the action of the cold-water malt-extract, and therefore the whole cupric reduction shown by the 10 per cent. solution after plain fermentation is not to be calculated as combined maltose. There is, on the contrary, every reason to believe that it is mainly due to gallisin, and it will be seen that consistent results are to be obtained if this view be adopted.

We assumed the residual cupric-reducing value of 1·46 to be due to gallisin, and 1·46 × 10 ÷ 1·01 = 14·4 per cent. of gallisin. This amount would cause a deviation in a 200-millimetre tube of 1·44 × 4·85 = 6·98 Soleil-Ventzke-Scheibler divisions, or 1·44 × 1·67 = 2·40 Laurent degrees.

The deviation observed after re-fermenting the fermented solution which had been treated with malt-extract was 22·8 Soleil-Ventzke-Scheibler divisions, or 7·84 Laurent degrees, and this, less the deviation due to gallisin, was 22·28 − 6·98 = 15·3 Soleil-Ventzke-Scheibler, or 5·26 Laurent degrees, and either (15·3 × 10 ÷ 11·56) or (5·26 × 10 ÷ 3·98) equal 13·2 per cent. of dextrin.

* Made by intimately mixing 100 grammes of finely-ground pale malt with 250 c.c. of water, and allowing the mixture to stand for twelve hours, giving it an occasional stir. The clear filtrate is used (*vide* Morris and Moritz, "Text-book of Brewing," pp. 477-480).

The matter remaining after treatment with malt-extract and re-fermentation amounted to 30 per cent., and as 27·6 per cent. was shown to be gallisin and dextrin, 2·4 per cent. was left unaccounted for.

The total matter which had disappeared during the first fermentation was 43·7 per cent., and deducting from this the 11·3 per cent. of cane sugar, 32·4 per cent. of reducing sugars were fermented. These consisted of a mixture of maltose and dextrose, having a cupric-reducing value of 6·106 (original K_{100} 7·65 – residue K_{100} 1·544), which equals 1·88 grammes of CuO to 1 gramme of substance. The deviation in a 200-millimetre tube equal to this 32·4 per cent. of substance was :—

	Soleil-Ventzke-Scheibler Divisions.	Laurent Degrees.
Original deviation	53·60	18·44
Deviation after first fermentation...	31·20	10·73
	22·40	7·70
Deviation corresponding to cane sugar estimation	4·34	1·49
	18·06	6·21

And either ($18·06 \times 10 \div 32·4 = 5·57$, divisor Soleil-Ventzke-Scheibler) or ($6·21 \times 10 \div 32·4 = 1·91$, divisor Laurent) equal the deviation in a 200-millimetre tube due to 1 gramme of substance.

These values correspond to a mixture of 16·84 parts of maltose and 15·56 dextrose.*

SUMMARY.

	Per cent.	Soleil-Ventzke-Scheibler. Corresponding angle 200 millimetres. 10 per cent. solution.	Corresponding K_{100} c.c., 10 per cent. solution.
Cane sugar	11·30	4·34	—
Maltose	16·84	13·50	2·30
Dextrose...	15·56	4·74	3·82
Combined maltose	0·61	0·49	0·08
Gallisin	14·40	6·98	1·46
Dextrin	13·20	15·26	—
Difference = unfermentable by plain fermentation	4·01	8·92†	—‡
Ash	1·34	—	—
	77·26	54·23§	7·66‖

Moisture = 22·74 per cent.

* From the cupric-reducing value :

$$2·46x + 1·37\,(1-x) = 1·88, \text{ and } x = \frac{51}{109} \text{ dextrose.}$$

From the rotation :

$$3·05x + 8·02\,(1-x) = 5·57, \text{ and } x = \frac{245}{497} \text{ dextrose.}$$

† Angle disappearing on degrading and refermenting.
‡ K taken account of already as combined maltose.
§ Compare with original angle 200 millimetres.
‖ Compare with original K_{100} c.c.

Discussion.

The Chairman (Mr. A. H. Allen) invited discussion, with special reference to the question of refermentation after the addition of malt-extract, and to the authors' assumption that gallisin was a substance of definite composition, possessing a definite reducing power and optical activity.

Dr. Sykes said that in all starch conversions, whether effected by acid or by diastase, undoubtedly certain bodies were present which could not be fermented by yeast alone, but which under the combined influence of yeast and diastase were readily fermentable, a fact utilized by the spirit or vinegar manufacturer, who did not boil his wort, and consequently preserved his diastase intact during the fermentation. In a glucose determination the removal of these bodies was, as mentioned in the paper, secured by fermenting the once fermented solution after the addition of malt-extract.

Dr. Dyer said he considered that, in dealing with this subject, a mistake had often been made in treating treacle, or any form of sugar syrup—a mixture of cane sugar and invert sugar—as an entity. Glucose syrup, however, for all practical purposes, could be so treated. Certain figures could be assumed as being safe outside limits to take for commercial glucose syrup, but no figures at all could be taken as representing constants for treacle or golden syrup.* Probably, indeed, some of the mixtures which had to be dealt with were not mixtures of glucose syrup with what would be called golden syrup or treacle, but consisted of commercial glucose syrup with the addition of crude sugar of some kind dissolved in water. In such cases the cane sugar was far higher in proportion to the invert sugar than would be the case in ordinary treacle or inverted syrup made from sugar. With the assistance of Mr. Sydney Steel, and from information kindly furnished to him by other chemical friends with experience in glucose syrup, he had arrived at the conclusion that it was quite safe to assume certain constants for glucose syrup: not exactly average figures, but figures representing the outside limits likely to be met with in the glucose syrup used for these mixtures—figures which would, if anything, give to the mixer the benefit of any doubt. The following were the figures assumed for glucose syrup:

Specific rotatory power $-[\alpha]_D$	$+113{\cdot}0$
Cupric oxide reducing power ("K value") ...	42

Where a polarimeter was used, which was graduated, not for angles, but for percentages of sugar, the rotation for "normal weight" dissolved in 100 c.c. of water was +170 divisions. Occasionally samples of glucose syrup were met with having a higher rotation, but such cases were exceptional. From polarization at 20° C., before and after inversion (by Herzfeld's method), and determination of the copper reducing power, the percentage of glucose syrup was calculated from the following formula, in which R stands for the specific rotatory power $[\alpha]_D$, at 20° C., of the uninverted sample, S for the + rotation due to the sucrose present, and K for the copper reducing power:

$$\text{Percentage of glucose syrup} = \frac{0{\cdot}206K + (R - S)}{1{\cdot}217}.$$

* Mr. Jones, in his paper on p. 87 of this number, suggests that, *after inversion*, sugar syrup may be regarded as an "entity." This materially reduces the liability to error through the assumption of a constant composition, but appears less satisfactory than the mode of calculation described in this paragraph.—B. D.

If a polarimeter reading percentages were used, and the observation made on "normal weight" of the syrup, R being the "percentage" reading before inversion and S the percentage of sucrose, the formula was:

$$\text{Percentage of glucose syrup} = \frac{0{\cdot}31K + (R - S)}{1{\cdot}83}.$$

The method was substantially that described some time back by Mr. Boseley for the estimation of glucose in marmalade, differing mainly in that the syrup, and not the "solids" dissolved in it, was treated as the direct basis of calculation. Mr. Chapman, after making a number of analyses of syrups more or less on the lines of those detailed in the paper of Messrs. Matthews and Hyde Parker, had also applied to his results this simple mode of calculation, and an interesting comparison was thus afforded between this method and the more elaborate and more scientific determinations which took into account the percentages of the different individual carbohydrates present.

Mr. CHAPMAN said that, whilst the rotation and copper oxide reducing power obtained, after fermentation were assumed by the authors to be due to unfermented dextrose and lævulose, modern methods of sugar analysis were to a great extent based on the assumption (which was universally believed to be justifiable) that dextrose and lævulose were, under proper conditions, entirely fermentable, and he ventured to think that the numbers referred to should rather be regarded as due to certain unfermentable bodies allied to the carbohydrates, which were well known to exist in raw sugars and in invert sugar, and to be possessed of very little optical activity, but of considerable cupric oxide reducing power. In some analyses of raw sugar recently published by Mr. Glendenning, unfermentable residues, which undoubtedly did not consist of carbohydrates, being possessed of very little optical activity, but having a cupric oxide reducing power of about 30, were shown to exist in almost all raw sugars in the proportion of from 1 to 3 per cent. With regard to the question of gallisin, he thought it a pity that definite assumptions should be made in regard to so indefinite a substance. The substance called gallisin had been obtained some years previously by a method which offered practically no guarantee as to its purity, and a body somewhat resembling it had since been obtained from glucoses and other commercial carbohydrate mixtures; but he thought there was insufficient evidence to warrant its being regarded as a body of definite composition. He had, as Dr. Dyer had mentioned, made analyses of a number of samples of adulterated treacle, four of which gave the following results:

	No. 1.	No. 2.	No. 3.	No. 4.
Cane sugar	20·63	26·10	27·40	4·10
Invert sugar	10·80	12·00	23·60	—
Dextrose	6·10	22·00	11·40	31·70
Maltose	26·40	·40	—	7·50
Dextrin	12·95	8·98	2·50	34·30
Water	19·08	20·50	22·69	21·30
Ash	1·82	5·20	7·24	1·10
Undetermined	2·22	4·82	5·17	—
	100·00	100·00	100·00	100·00
Optical activity $[a]_D$ =	+ 77·3°	+ 45·2°	+ 24·6°	+ 99·01°

From a consideration of their carbohydrate constituents, he had concluded that they contained 52, 32, 13 and 87 per cent. of glucose syrup respectively; the comparatively simple method of calculation explained by Dr. Dyer gave 56·9, 27·3, 10·5 and 86·4 as the respective percentages of glucose syrup. He was now quite satisfied that the simpler method of dealing with the subject was quite sufficient for the practical purpose of determining the approximate percentage of glucose syrup in these mixtures.

Mr. BOSELEY said that he had employed the method referred to in his paper on "The Analysis of Marmalade" (ANALYST, xxiii., 123) in the analysis of a very large number of samples of jam, and had found it to work most admirably. In determining the cupric oxide reducing power in a jam, however, it was necessary to remember that in some fruits there was present a substance which was not sugar, but which, apart from the natural invert sugar of the fruit, was capable of reducing Fehling's solution, and the results published in 1898 were probably about 2 per cent. too high owing to the influence of this substance, of the presence of which, in sufficient quantity to affect the results, he was not at the time aware. He had examined a great many samples of glucose syrup, and had not found the rotation to vary largely. The figure he had adopted for "percentage" rotation (cane sugar = 100), viz., 166

(or Sp. R. P. $[\alpha]_D = +110°$),

was an average of between 200 and 300 samples obtained from different sources, the highest result obtained being 173

(or Sp. R. P. $[\alpha]_D = +115°$).

Mr. JULIAN L. BAKER said it seemed very unsatisfactory that in the determination of cupric reducing power the method of weighing as cupric oxide should still be indulged in. It was far more accurate, and was now almost universal—at any rate, amongst Continental chemists—to weigh as metallic copper. It made a great difference whether, in speaking of gallisin, the authors meant conversion products, or whether they meant the unfermentable residues of starch conversion. On degrading starch by means of malt-extract in the cold, a substance was obtained which was practically wholly fermentable; whereas, if the operation were conducted at a higher temperature, conversion products were obtained which were not completely fermentable. Some of these unfermentable products had been examined by Mr. Ling and himself, and had been found to consist of bodies having the formula $C_{12}H_{22}O_{11}$. Their composition thus resembled that of maltose, and they were non-crystalline and had a fairly high reducing power. He was at present working, together with Mr. T. H. Pope, upon a series of polysaccharides, which occurred largely in the vegetable kingdom, and among which was probably the body mentioned by Mr. Boseley. They were non-crystalline, and yielded definite sugars on hydrolysis. He had recently isolated one of these bodies from the ivory nut, having the composition $C_6H_{10}O_5$, which on hydrolysis yielded considerable quantities of mannose, in addition to a lævo-rotatory sugar, probably lævulose, and which, while reducing Fehling's solution, decomposed at high temperatures, a peculiarity not possessed by the other members of the series which he had examined.

Mr. ARCHBUTT recommended the method of estimating cuprous oxide recently described by Messrs. Caven and Hill before the Nottingham section of the Society of

Chemical Industry,* in which the precipitate was filtered on an asbestos mat in a Gooch crucible, and transferred, after thorough washing with boiling distilled water, to a flask containing a mixture of dilute sulphuric acid and standard potassium permanganate solution, the excess of permanganate being titrated with oxalic acid or hydrogen peroxide.

ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

FOODS AND DRUGS ANALYSIS.

The Determination of Cane-Sugar in Condensed Milk. L. Grünhut and S. H. R. Riiber. (*Zeit. Anal. Chem.*, 1900, xxxix., 19-36.)—This paper contains an account of the authors' critical examination of various methods of estimating cane sugar in the presence of milk sugar.

Of the methods based on reduction with Fehling's solution before and after inversion, they regard those in which hydrochloric acid inversion is used as of chief importance in the analysis of condensed milk.

They point out that, if a correct conclusion as to the amount of cane sugar is to be drawn from the increase in the reducing power after inversion, it is essential that the reductions before and after inversion must be made under exactly the same conditions, since every deviation, either in the concentration of the liquid or the length of time of heating, influences the amount of cuprous oxide deposited.

The only gravimetric methods known to them which fulfil this condition are those of Ost (ANALYST, xx., 259) and of Kjeldahl (ANALYST, xx., 227), and the former of these has been shown both by Ost himself and by Schmöger to be unsuitable for the determination of milk sugar. Pavy's volumetric method is not included in the investigation, since the readiness with which the ammoniacal solution undergoes alteration renders the results uncertain.

In applying Kjeldahl's method, however, in which a specially strong Fehling's solution is used and the liquid boiled for twenty minutes, there is the probability of a certain amount of decomposition of the cane sugar taking place, with the formation of decomposition products with strong reducing properties. G. Bruhns, in fact, has shown that this decomposition leads to considerable error (ANALYST, xxiii., 297).

A second source of error in the copper reduction methods in the analysis of condensed milk is that in the direct reduction too great an amount of cuprous oxide is separated, with the result that the milk sugar is overestimated. The authors' experiments show that the amount of copper reduced varies with the quantity of the milk taken. For example, a mixture of sixty parts of Passburg's dry milk (containing 21·38 per cent. of milk sugar) with forty parts of cane sugar yielded by direct

* *Jour. Soc. Chem. Ind.*, xvii. (1898), 124.

reduction in solutions of different degrees of concentration quantities of cuprous oxide corresponding to (1) 21·52 per cent. and (2) 23·20 per cent. of milk sugar.

This error depends not only on the relative proportion of milk and cane sugar, since this was the same in the above experiments, but also on the absolute quantity of sugar. In the first case a large quantity of the sugar solution was taken, and the result was nearly correct, whilst in the second much less was used and the milk sugar was too high.

The explanation given for this is that the milk sugar commences its reducing action as soon as the liquid begins to boil, and reduction is nearly completed before any decomposition of the cane sugar takes place and causes a secondary reduction. Thus, if there be much milk sugar present, so much of the copper solution is reduced that its subsequent action on the cane sugar is greatly weakened and the error will only be trifling.

The action of the solution on the cane sugar is greatest when no milk sugar is present. When both cane and milk sugar are present, less cuprous oxide is deposited than corresponds to the sum of the amounts from the two reactions.

Hence, the authors conclude that the only method of accurately estimating milk sugar in the presence of cane sugar by means of Fehling's solution is to construct an empirical table in which a correction is made for each absolute and relative amount of both sugars.

Another inherent error in these reduction methods is that it is not correct to simply calculate the quantity of cane sugar from the difference between the amounts of copper reduced by the milk sugar before inversion, and by the milk sugar and the invert sugar after inversion. For example, in the case of the prepared condensed milk mentioned above, this difference for 0·375 gramme of the mixture amounted to 0·26695 gramme of copper. This corresponded to 0·13517 gramme of invert sugar, or 34·24 per cent. of cane sugar, instead of the theoretical 40 per cent.

Somewhat better, though still incorrect, results are obtained by calculating both amounts of copper into the corresponding quantities of invert sugar and taking the difference. In this way the cane sugar (40 per cent.) in the above mixture was found to be 38·64.

This deviation is due, in the main, to the fact that the products of the reduction of two sugars causing simultaneous reduction cannot be directly added together. Kjeldahl believed that he had discovered the law underlying this reaction (*Zeit. anal. Chem.*, xxxv., 347 and 646), and gave directions for its application to the determination of two kinds of sugar. The authors, however, have been unable to obtain satisfactory results by using Kjeldahl's directions, and in the case of their prepared condensed milk found −14·23 per cent. of milk sugar and +62·81 per cent. of cane sugar.

Summing up the results of their experiments on the reduction methods, they have arrived at the conclusion that it is not possible by their means to effect an accurate determination of cane sugar in condensed milk.

A second class of methods is based on the polarization of the solution before and after inversion, and the calculation of the cane sugar with the aid of the Clerget formula.

In order to obtain correct results in this way, the authors adopt several precautions. By treating the condensed milk with boiling water and allowing the solution to cool, they state that the influence of the multirotation of the milk sugar is completely obviated. They have not met with the difficulty experienced by Richmond and Boseley (ANALYST, xviii., 141 and 171), who found that the specific rotatory power of cane sugar was considerably altered by heating the solution at 100° C.

In making a correction for the volume of the casein and fat precipitated by means of lead acetate or of mercuric nitrate as proposed by Wiley, they make use of the method of double dilution (*cf.* ANALYST, xxi., 182). For the calculation of the results they have found Herzfeld's modification of Clerget's formula (*Zeit. anal. Chem.*, xxxv., 717) more satisfactory than the original formula. Using the latter, they found 39·12 per cent. of cane sugar in their experimental mixture, whilst with the former they obtained 39·39 per cent.

The empirical factor (0·962), which has been prescribed by the *Bundesrath* for the correction of the volume, is objected to on the ground that it is only applicable to preparations of one particular chemical composition, so that when there is any considerable variation from that type the results are much less satisfactory.

C. A. M.

Gerber's Process for the Estimation of Fat in Butter. **J. Werder.** (*Chem. Zeit.*, 1899, xxiii., 1028.)—As originally devised, this process did not give trustworthy results, but it has recently been improved, and is now fit to rank as a regular laboratory method. In a small capsule, which forms the stopper of a specially graduated tube, 5 grammes of the well-mixed sample are weighed out; it is then put in position, and the tube is filled with 1 c.c. of amylic alcohol and 20 c.c. of sulphuric acid (specific gravity 1·50). After agitation and separation the volume of fat is read off. Double tests carried out on twelve samples of market butter showed maximum differences one from another of 0·42 per cent., and the mean figures, in comparison with those given by the Soxhlet process, showed maximum differences of +0·30 and −0·45 per cent., or an average difference of ±0·27 per cent. Applied to two remelted butters, the yields were between 99·6 and 100·1 per cent. F. H. L.

Contribution to our Knowledge of the Composition of Hens' Eggs. **A. Juckenack.** (*Zeit. für Untersuch. der Nahr. und Genussmittel*, ii., 1899, 905.)—On incinerating yolk of egg as a preliminary to the determination of the inorganic constituents, a portion of the phosphoric acid becomes reduced to phosphorus, and is therefore lost, owing to the bases being in insufficient proportion to form metaphosphates. The difficulty may, however, be overcome by the addition of alkaline carbonate and nitrate before incinerating. In white of egg, and in the combined white and yolk, mixed in the proportion in which they occur in whole eggs, the bases are present in excess, and correct results are consequently obtainable without such addition.

The following results are given:

I.	Yolk	1·279 per cent. phosphoric acid.	
II.	White	0·031	,, ,,
III.	Whole eggs:		
	(*a*) By direct determination ...	0·443	,, ,,
	(*b*) By calculation from I. and II.	0·455	,, ,,
IV.	Average phosphoric acid contents of one egg (16 grammes yolk and 31 grammes white.)	0·214 gramme.	

Table showing the Various Combinations and Proportions in which the Phosphoric Acid exists in Yolk of Egg.

Total phosphoric acid in 100 grammes, 1·279 grammes.

Soluble in boiling alcohol, 0·823 grm. = 9·35 gramme distearyllecithin.		Insoluble in boiling alcohol, 0·456 gramme.	
Extractable direct by ether from the yolk, 0·478 gramme = 5·42 grammes free distearyllecithin.	Dissolved by alcohol after extraction by ether, 0·345 gramme = 3·93 gramme distearyllecithin combined with vitellin.	As nucleïn, 0·178 grm.	As insoluble phosphate or compounds of phosphoglyceric acid, 0·278 gramme.

The cholesterin in yolk of egg was also determined, the result showing 0·91 per cent., corresponding to 1·92 per cent. in the dry substance. H. H. B. S.

The Reliability of the Glycerin titration Method for the Determination of Boric Acid in Preserved Meats, and the Separation of Boric Acid from Borax. A. Beythien and H. Hempel. (*Zeit. für Untersuch. der Nahr. und Genussmittel*, ii., 1899, 842-851.)—The authors have carried out experiments to test the accuracy of the glycerin titration method, which depends upon the fact that if a solution of boric acid be neutralized, using methyl orange as indicator, and then glycerin added, the solution reacquires an acid character, and can be again titrated with alkali, phenolphthalein being used as indicator. The authors followed in general the directions given by Jörgensen. The results affirmed the reliability of the process, and showed in particular that no appreciable loss takes place, either when the acidified boric acid solution is heated, or when the alkaline residue left after evaporation is ignited. The maximum loss in the experiments amounted to 1·66 per cent. of the quantity taken.

The authors also tested the process in its application to the examination of preserved meats. Weighed portions of chopped beef were mixed with measured quantities of solutions of boric acid of known strength. After the addition of water, solution of soda was added until a decided alkaline reaction was produced. The mixture was then warmed for several hours and filtered. This procedure was carried out three times, after which the various filtrates were mixed together and evaporated to dryness. The residue was then incinerated, the ash taken up with sulphuric acid, the solution gently heated to expel carbonic acid, cooled and

neutralized. Twenty-five c.c. of glycerin were then added, and the mixture titrated with standard soda. The greatest differences were: −5·06 and +1·67 per cent. of the boric acid taken.

Aspirin. F. Goldman. (*D. Pharm. Ges. Ber.*, 1899, ix., 232; through *Chem. Zeit. Rep.*, 1899, 340.)—Aspirin is a product of the action of acetic acid upon salicylic acid, its formula being $C_6H_4(O.COCH_3)COOH$. Its melting-point is 135° C. When 0·5 gramme is boiled with 10 c.c. of a 10 per cent. solution of sodium hydroxide, a clear liquid should result, in which an excess of dilute sulphuric acid produces a temporary violet colour, and precipitates the salicylic acid, which can be filtered off and identified by its melting-point and its reaction with ferric chloride. The filtrate from the salicylic acid has an odour of acetic acid, and on boiling with alcohol and sulphuric acid gives that of acetic ether. Aspirin should not contain any free salicylic acid; to detect it, 0·1 gramme is dissolved in 5 c.c. of alcohol, diluted with 20 c.c. of water, and 1 drop of ferric chloride solution is added; no violet colour should appear. F. H. L.

ORGANIC ANALYSIS.

Estimation of Benzene Vapour in Illuminating Gas. O. Pfeiffer. (*J. Gasbeleucht*, 1899, xlii., 697; through *Chem. Zeit. Rep.*, 1899, 333.)—The author suggests a modification of Harbeck and Lunge's nitration process (ANALYST, 1898, xxiii., 101) which makes it far more simple to carry out. The gas is measured and nitrated in a large separating funnel holding 3 or 4 litres; it is filled through the stopper-hole by displacement, and 10 c.c. of the mixed nitrating acids are introduced through the ordinary exit tube.* The acids are made to spread over the internal surface, and are allowed to act for an hour, when the whole of the benzene will be absorbed. The vessel is then rinsed out with 100 c.c. of water, which is brought into a small separating funnel, and neutralised with crystallised sodium hydroxide (40 grammes). It is extracted two or three times for five minutes with quantities of 50 c.c. of ether, each of which is afterwards washed two or three times with 10 c.c. of water to remove some brown substance. The solvent is finally evaporated, the residue taken up in a little anhydrous ether, filtered through freshly calcined sodium carbonate into an evaporating basin, and dried over sulphuric acid—it should neither be warmed nor placed in vacuo. To convert the weight (g) of dinitrobenzene into the percentage by volume of benzene vapour, the author gives the following simplified formula; C is the capacity of the measuring vessel, t the temperature, and b the height of the barometer:

$$\frac{36080}{C} \times g \times \frac{273+t}{b}.$$

Having a complete analysis of the gas, and knowing its specific gravity, it is also possible to calculate the proportion of benzene. Calling the specific gravity of

* The stopcocks should be "lubricated" with strong sulphuric acid.

the gas S, the specific gravity of the "heavy hydrocarbons" (C_nH_m) s, and the total volume of the latter v, we have:

$$s = 100S - \left(CO \times 0{\cdot}9621 + CH_4 \times 0{\cdot}5530 + H \times 0{\cdot}0692 + N \times 0{\cdot}9701 + CO_2 \times 1{\cdot}5197 \frac{1}{C_nH_m}\right)$$

whence the percentage by volume of benzene vapour becomes:

$$\frac{(s - 0{\cdot}9674)v}{1{\cdot}7367}.$$

As an example of this method, the author quotes a sample of gas in which the proportion of benzene was calculated to be 1·57 per cent., and that of ethylene 1·83 per cent.; by direct analysis the benzene was returned at 1·52 per cent., and the ethylene 1·88 per cent. F. H. L.

The Volumetric Estimation of Quinones of the Benzene Series. A. Valeur. *Bull. Soc. Chim.*, 1900, xxiii., 58-61.)—This method is based on the reduction of the quinones by hydriodic acid.* When the latter is replaced by an equivalent quantity of hydrochloric acid and potassium iodide, the reaction takes place in accordance with the equation:

$$C_6H_4O_2 + 2HCl + 2KI = C_6H_6O_2 + 2KCl + I_2.$$

In the determination a quantity of the pure dry quinone, sufficient to liberate from 0·20 to 0·50 gramme of iodine is dissolved in a little 95 per cent. alcohol. To this solution is added a rapidly-prepared mixture of 20 c.c. of a 10 per cent. solution of potassium iodide with 20 c.c. of concentrated hydrochloric acid previously diluted with an equal volume of 95 per cent. alcohol and cooled. The liberated iodine is titrated with standard thiosulphate, and the corresponding amount of quinone calculated.

The following results show the accuracy of the method:

	Toluquinone, $C_6H_3(CH_3)O_2$.		Thymoquinone, $C_6H_2(CH_3)(C_3H_7)O_2$.	
	I.	II.	I.	II.
Weight of substance, gramme	0·2057	0·2707	0·2130	0·1663
Iodine liberated ,,	0·4290	0·5629	0·3311	0·2574
Iodine per cent.	208·5	207·9	155·4	154·7
	(Theory 208·2)		(Theory 154·8)	

It is necessary to mix the hydrochloric acid and the potassium iodide, and not to add them separately to the quinone, since the acid would immediately react on the quinone, and the iodide would cause partial oxidation. This method appears to be applicable to the majority of true quinones. It can be used to determine their solubility in different solvents, which is not easily determined by other methods, on account of the volatility of the quinones and the difficulty of drying them without loss.

It can also be employed for the determination of quinones in somewhat unstable combinations, such as the phenoquinones and quinhydrones. In the case of

* Reduction with HI yields a mixture of hydroquinone and quinhydrone; from what is stated in the paper it appears that with KI and HCl the reduction is carried a step further, and gives only hydroquinone.

ordinary quinhydrone the following percentages of iodine were obtained: 116·6, 116·3 and 116·1, whilst the calculated percentage for $C_6H_4O_2 \cdot C_6H_6O_2$ is 116·5. The author therefore concludes that this compound, contrary to the view of Wichelhaus, results from the union of equal molecules of quinone and hydroquinone. C. A. M.

The Properties of the Oils of Lemon, Bergamot, and Orange. A. Soldaini and E. Berté. (*Boll. chim. farm.*, 1899, xxxviii., 537; through *Chem. Zeit. Rep.*, 1899, 323.)—Some constants of these three oils in the pure state are given in the annexed table:

	Lemon.	Bergamot.	Orange.
Specific gravity at 15°	0·854-0·860	0·882-0·886	0·847-0·853
Rotatory power at 20° (100 millimetres tube)	56-66°	8-20°	96-98°
Boiling-point at ordinary pressure	171-172°	—	173-174°

Lemon Oil.—In lemon oil the proportion of citral should not be below 6·5 per cent.; and when 20 grammes are fractioned at a pressure of 20 to 30 millimetres, the first 10 c.c. of the distillate should have as high a rotatory power as the original oil. The presence of orange oil is shown (*a*) by a yellow colour when a drop of the sample is mixed with 15 or 20 drops of brominated chloroform, (*b*) by a yellow flocculent precipitate instead of a white crystalline one when sodium bisulphite solution is added. To determine the percentage of citral, a 5 c.c. pipette graduated in fortieths is required, and also a pear-shaped flask, the neck of which has the same diameter as the pipette, and which has a lateral tube bent upwards at a right angle to carry a funnel. Five c.c. of the sample are run from the pipette into the flask, 25 c.c. of a solution of potassium bisulphite containing an excess of sulphur dioxide are introduced, the lower end of the pipette is connected to the neck, the whole is shaken and warmed for twenty minutes on the water-bath, then cooled and warmed again for five minutes. When finally cold, the volume of oil still remaining liquid is read off in the pipette by adding water through the side funnel; and the difference between it and the 5 c.c. taken represents the volume of the aldehyde.

Bergamot Oil.—The proportion of linalyl acetate usually varies between 21 and 22 per cent. When 15 c.c. are fractioned at the above pressure, the first 5 c.c. of the distillate should have a rotatory power 2½ times as great as the oil itself; and the next 9·5 c.c. should be almost inactive. Evaporated on the water-bath, the residue should be between 5 and 6 per cent. The oil should be soluble in ½ vol. of 90 per cent. alcohol, and the clear solution should not be rendered turbid on dilution. Schiff's reagent for aldehydes should fail, or at most give a faint tint in half an hour; an immediate colour, or a strong red in half an hour, indicates lemon oil. To estimate the linalyl acetate, 1·5 grammes are saponified with an excess of seminormal alcoholic potash, diluted with a little 80 per cent. spirit, and titrated with seminormal sulphuric acid and phenolphthalein; the volume of alkali multiplied by 0·09775 (mol. wt. of the ester, 195·5) gives the acetate.

Orange Oil.—When 20 c.c. are fractioned at a pressure of 10 or 20 millimetres, the distillate should have a rotatory power from 1° to 3° higher than the original. Schiff's reagent should give no colour. F. H. L.

Determination of the Solidifying-point of Fatty Acids. I. Freundlich. (*Chem. Zeit.*, 1899, xxiii., 1014.)—The ordinary Dalican process for determining the exact solidifying-point of fatty acids is not quite accurate, for the temperature to which the thermometer finally rises is partly dependent on that to which it was made to fall during the stirring of the fat. The following modified way of carrying it out leads to absolutely concordant results, or at the worst to differences of 0·05 to 0·1° C. At the lowest temperature above the expected solidifying-point at which the sample round the thermometer bulb remains perfectly liquid, the thermometer is moved quickly two or three times backwards and forwards through the fat, and the mercury is observed; if it falls sharply, the operation is repeated until the column remains constant for thirty or forty seconds; then the sample is stirred fifteen or twenty-five times, and if the mercury falls during the agitation and rises immediately afterwards to a maximum at which it stands unchanged for three or five minutes, that maximum is the true solidifying-point. The great thing to avoid is too prolonged stirring. F. H. L.

The Becchi and Halphen Colour Reactions for Cotton Oil. P. N. Raikow and N. Tscherweniwanow. (*Chem. Zeit.*, 1899, xxiii., 1025.)—At the present time there exist some ten different modifications of the Becchi test for cotton oil; and statements as to its utility and the best proportions for its several ingredients vary enormously. Benedikt and other authorities question whether cotton oils do not occur which cause no reduction of the silver nitrate at all; this, however, seems problematical, unless the samples have been specially treated in order to prevent their giving the Becchi reaction. It is not possible to render cotton oil indifferent to the Becchi test by blowing air through it in the cold; nor can the same object be attained by repeated extractions with alcohol, the latter fact being in contradiction to Benedikt's assertion that the true cause of the reaction is an aldehyde-like body which is readily soluble in spirit. Even the fatty acids of cotton oil after alcoholic saponification and liberation from their barium salts, or after aqueous saponification and liberation from the sodium salts, give the test, while the small proportion of unsaponifiable matter in the original oil does not give it. Treated with ordinary steam even for long periods of time, cotton oil still retains its usual properties; but superheated steam, or a simple heating of the oil to between 210° and 220°, quickly destroys its reducing power, which also vanishes more slowly at 150°, the residual material being almost unaltered, except that its colour is slightly darkened, and that it possesses a faint burnt odour. Precisely the same remarks apply to the Halphen test: one hour at 150° hardly affects the reaction, five hours reduce its intensity to one-half, ten hours to one-third; in time, probably, it would fail altogether. [*Cf.* Holde and Pelgry, ANALYST, 1899, xxiv., 214.]

After elaborate experiments, the authors find that the details of the Becchi test are most important: the proportion of silver nitrate to the oil, the proportion of free acid to the silver, ought to be kept uniform; and they decide that the method adopted by the Italian Commission (*cf.* ANALYST, 1895, xx., 222, last paragraph) should be taken as the standard. The latest modification of the Becchi test described by

Tortelli and Ruggeri (ANALYST, 1898, xxiii., 179) was not examined; in comparison with the more certain and very delicate Halphen reaction, it is too complicated to be generally useful.

It appears to be universally admitted that the Halphen reaction (ANALYST, 1897, xxii., 326; 1898, xxiii., 131) is better than Becchi's; the only points of divergence are its degree of delicacy and the best method of carrying it out. The actual temperature employed is only a matter of convenience; a cherry-red colour is produced in the cold by bright sunlight in three hours. The part played by the amylic alcohol is obscure, but it does not serve simply to retain the carbon disulphide in the hot liquid; it should, therefore, not be omitted. A faint Halphen red is manifested when the free sulphur is left out of the regular mixture; an excess of sulphur is useless, and in the testing of oils containing only a little cotton oil, it tends to decrease the delicacy of the reaction. The present authors accordingly reject Soltsien's proposals; there is no objection to the use of plain water instead of the brine bath, but in other respects Halphen's prescription should be retained in its integrity. The behaviour of olive, walnut, linseed, poppy, and arachis oils towards the test has been studied under various conditions; neither by prolonged heating on the water-bath nor in sunlight did they respond. In pale-coloured oils 0·5 per cent. of cotton oil can be readily detected, and this proportion may be taken as its limit of sensitiveness.

F. H. L.

The Determination of the Bromine Absorption of Oils. P. C. McIlhiney. (*Journ. Amer. Chem. Soc.*, 1899, xxi., 1084-1089.)—In a former communication (ANALYST, xix., 141) the author described a process for the determination of this constant in which a distinction was made between the bromine addition and bromine substitution values.

He has now simplified his process by making use of the iodometric method of Schweitzer and Lungwitz (*Journ. Soc. Chem. Ind.*, 1895, 130), and shortening the time in accordance with the fact that the addition of bromine to fats is practically instantaneous (ANALYST, xx., 146).

In the modified process a weighed quantity of the oil is dissolved in 10 c.c. of carbon tetrachloride in a stoppered bottle and 20 c.c. of one-third normal bromine in carbon tetrachloride added. Simultaneously a blank determination is made, and subsequently titrated with standard thiosulphate to determine the strength of the bromine solution. After the lapse of one or two minutes, 20 to 30 c.c. of a 10 per cent. solution of potassium iodide are introduced, the bottle shaken to insure the absorption of the bromine and hydrobromic acid, and the iodine titrated with deci-normal thiosulphate.

After the titration, 5 c.c. of a neutral 2 per cent. solution of potassium iodate are introduced, and the amount of iodine liberated, which is equivalent to the hydro-bromic acid formed, is titrated, and gives the bromine substitution figure.

In order to prevent a loss of bromine or hydrobromic acid on removing the stopper to introduce the potassium iodide, a piece of wide indiarubber tubing is slipped over the neck of the bottle, thus forming a well round the stopper. The

potassium iodide is poured into this well and the stopper slightly opened, preferably after the bottle has been cooled in ice to create a partial vacuum in the interior. The following figures were thus obtained with various representative oils. The difference between the figures given in the last column and 1·000 are intended to represent the degree of substitution which occurred in the determination of the Hübl value:

	Hübl Value.	Bromine Value calculated from Hübl Value.	Bromine Absorbed per Cent.	Bromine Addition Value.	Bromine Substitution Value.	Calculated Bromine Value divided by Bromine Addition Value.
Raw linseed oil, several years old	157·3	99·1	99·2	92·0	3·6	1·077
„ „ „ average of seven samples	183·8	115·7	112·0	106·6	2·7	1·083
Boiled linseed oil, average of eight samples	—	—	109·5	103·0	3·2	
Third run rosin oil	63·9	40·3	92·3	7·7	42·3	5·231
"Java" boiled rosin oil	73·3	46·2	101·9	8·3	46·8	5·685
Menhaden oil, average of three samples	174·9	110·2	110·6	95·6	7·5	1·154
Maize oil, average of three samples	—	—	75·8	72·9	1·5	
Cotton-seed oil	—	—	65·8	62·2	1·8	
Turpentine			266·1	166·1	50·0	
Ceylon cocoanut oil			5·36	4·7	0·33	
Tallow rendered in laboratory			24·0	21·48	1·26	
Hard paraffin			3·55	1·43	1·06	
Black rosin	—	—	135·4	5·4	65·0	

The advantages claimed for this method are that the bromine solution is readily prepared and does not change on keeping, that it is exceedingly rapid, and that it distinguishes between the halogen absorbed by addition and by substitution.

C. A. M.

INORGANIC ANALYSIS.

Bromine and Alkali Hydroxides as a Test for Copper. D. Vitali. (*Boll. Chim. Farm.*, 1899, xxxviii., 665; through *Chem. Zeit. Rep.*, 1899, 349.)—It is well known that when a solution of copper sulphate is mixed with potassium or sodium hydroxide, and an excess of bromine water is added, the original blue precipitate soon becomes black. This substance consists of copper dioxide; it evolves oxygen on warming or on treatment with sulphuric acid, and it gives off chlorine with hydrochloric acid. The reactions proceed thus:

$$CuSO_4 + 2NaOH = Cu(OH)_2 + Na_2SO_4.$$
$$2NaOH + Br_2 = NaBr + NaBrO + H_2O.$$
$$Cu(OH)_2 + NaBrO = CuO_2 + H_2O + NaBr.$$

Other oxidizing agents, such as hydrogen peroxide or manganese dioxide, also convert copper hydroxide into dioxide.

By evaporating 1 c.c. of a solution containing copper to dryness, treating the residue with the yellow mixture of alkali and bromine, a black precipitate will be produced if the proportion of copper was 1 : 100,000. If the residue is more simply moistened with saturated bromine water and again dried, a black deposit is obtained, consisting of $CuBr_2$, which becomes blue on being touched with water. This test will show copper in 1 c.c. of a 1 : 1,000,000 solution, and it is therefore more sensitive than sulphuretted hydrogen or potassium ferrocyanide. The black bromide is rendered more visible when moistened with strong sulphuric acid.

Other metallic salts yield black precipitates with alkali hydroxides and bromine, *e.g.*, $Co_2(OH)_6$, $Ni_2(OH)_6$, MnO_2H_2O. Mercurous salts give a yellow precipitate of mercuric oxide. Bismuth salts give a reddish-brown precipitate of Bi_2O_4. Lead salts in the cold finally yield an orange-yellow precipitate of lead oxybromide, which changes into peroxide on warming. F. H. L.

The Separation of Tungsten and Molybdenum. F. Ibbotson and H. Brearley. (*Chem. News*, lxxxi., 13-15.)—Two modes of procedure are recommended :

(*a*) The tungsten and molybdenum are precipitated together by lead acetate, the precipitate washed slightly with hot, very dilute acetic acid and ignited. The ignited precipitate is then dissolved in concentrated hydrochloric acid, using about as many c.c. of acid as there are centigrammes of the lead salts. Two or three times the bulk of boiling water is then added, the solution boiled, allowed to settle, filtered by decantation through a small pulp filter, the precipitate redissolved in about half the quantity of acid previously used, reprecipitated with water, and filtered through the same pulp. The molybdenum is determined in the filtrate as $PbMoO_4$. The greater the amount of acid used to dissolve the lead salts, the more water is required for the complete precipitation of the tungstic acid, and the greater the dilution, the greater the liability to contamination with molybdic acid.

(*b*) When the quantity of tungsten is small in comparison with that of the molybdenum, the former is not completely precipitated. Under these circumstances the following procedure is recommended : A few drops of nitric acid are added to the hydrochloric acid solution of the lead salts, which is then evaporated to a pasty consistence. The mass is diluted with 200 to 300 c.c. of dilute hydrochloric acid (1 : 3), the solution boiled, and the tungstic acid filtered off. The precipitate adhering to the sides of the vessel can be removed by dissolving in a few drops of dilute ammonia, which may then be absorbed with a piece of filter-paper, and the latter ignited with the precipitate. H. H. B. S.

Estimation of Tellurium Dioxide in Presence of Haloid Salts. F. A. Gooch and C. A. Peters. (*Zeits. anorg. Chem.*, 1899, xxi., 405.)—Brauner has shown (*J. Chem. Soc. Trans.*, 1891, 238) that tellurous acid cannot be determined by oxidation with permanganate if it be dissolved in hydrochloric acid ; and he has also stated that if the oxidation take place in sulphuric acid solution a small correction is

necessary, which is not required when the oxide is dissolved in alkali. If, therefore, the tellurous acid is dissolved in sodium hydroxide, and if the final titration between the oxalic acid and permanganate is carried out under those conditions which are desirable to avoid the interfering action of hydrochloric acid—*i.e.*, presence of manganous chloride—tellurium dioxide can be accurately determined in the manner indicated, even if it be accompanied by chlorides. About 0·1 gramme of TeO_2 is dissolved in a little sodium hydroxide, and mixed with permanganate solution (standardized on ammonium oxalate) till it remains pink; the liquid is heated, and 5 c.c. more of 1 : 1 sulphuric acid than are needed to neutralize it are added; excess of standard ammonium oxalate is introduced to destroy the higher oxides of manganese and the excess of permanganate; and finally the excess of oxalate is titrated with permanganate. If the proportion of hydrochloric acid is quite small, *e.g.*, such as is produced by the decomposition of the original tellurium chloride, addition of manganous chloride is not necessary; but it is better always to employ it (0·5 to 1 gramme), as the last titration can then be conducted in the cold.

In the presence of a bromide fairly satisfactory results can be obtained, provided that the excess of sulphuric acid is not greater than 5 c.c. of a 12·5 per cent. acid, that sufficient manganous chloride (0·5 to 1 gramme) is used, and that the liquid is titrated at ordinary temperatures.

The process is obviously not available in the presence of an iodide. The method described by Norris and Fay (ANALYST, 1898, xxiii., 249) gives excellent results. Instead, however, of titrating the iodine liberated from the iodide and sulphuric acid by means of thiosulphate, it can be determined with decinormal arsenious acid, this modification possessing the advantage that the arsenic serves also to standardize the original permanganate used to oxidize the tellurium dioxide. About 0·5 or 1 gramme of potassium iodide dissolved in 100 c.c. of water is added to the alkaline solution of the tellurium, then standard permanganate is run in till the green colour disappears (about 30 c.c. of the decinormal liquid per 0·1 gramme of TeO_2); next a few c.c. of sulphuric acid are added to clarify the liquid, and the free iodine is titrated in presence of potassium bicarbonate. It is important to have a sufficient excess of iodide, and it is perhaps better to run in some of the arsenious acid before acidifying with sulphuric acid. All the examples quoted by the authors are reasonably satisfactory, and they agree closely with theory when the atomic weight of tellurium is taken at 127. F. H. L.

Determination of Sulphur in Bitumens. S. F. and H. E. Peckham. (*Journ. Amer. Chem. Soc.*, vol. xxi. [9], pp. 772.)—The authors have modified their deflagration method by taking such an amount of the assay as will correspond to about 0·2 gramme of bitumen, and mixing it with 15 grammes each of pure, dry sodium carbonate and potassium nitrate, the mixture being then deflagrated in small successive portions in a 2-ounce platinum crucible at dull red heat. No blast lamp is necessary, the fusion being complete without; the excess of flux reduces the violence of the operation and the risk of loss by spattering.

The mass is dissolved by immersing the crucible in water over night, and the silica, iron, alumina and sulphuric acid determined in the solution in the usual manner. C. S.

REVIEWS.

DETERMINATION OF RADICLES IN CARBON COMPOUNDS. By Dr. H. MEYER (Prague). Authorized translation, by Dr. J. BISHOP TINGLE (Chicago). New York: J. Wiley and Sons; London: Chapman and Hall, 1899. Price 4s. 6d.

This small book is a valuable addition to literature, containing as it does within the limits of 120 pages much useful information and many valuable hints. Its scope is broader than might be inferred from the title; it really deals with the principles of methods for the proximate analysis of organic compounds. Although it might appear at first sight to appeal more particularly to the investigator in organic chemistry than to the technical analyst, it must not be forgotten that the problems with which both are confronted very frequently differ only in the form in which they are presented. Methods which depend on the determination of certain atomic groups and radicles find in point of fact frequent application alike in purely scientific investigations and in the technical analysis of organic products. Some of these have, in their technical application, become quite stereotyped, and are perhaps too often used by analysts without regard to the scientific principles on which they are based. The use of such terms as "acetyl value," "iodine value," convenient though they may be, are doubtless largely responsible for this, yet the analyst should in all cases consider what he is actually measuring. Thus, that the acetyl value is a measure of displaceable hydrogen in the groups – OH, = NH, etc., and that the iodine value indicates the presence of unsaturated compounds.

To briefly summarize the contents of the book, it describes methods for the determination of the following groups: $-OH$, $-OCH_3$, $-OC_2H_5$, $-COOH$, $=CO$, $-NH_2$, $-CN$, $-CONH_2$, $=NH$, $=NCH_3$, the diazo, hydrazine, iodoso, iodoxy, and peroxide groups. Instructions are also given for the determination of the "iodine value."

Methods for the determination of the basicity of acids, and for the introduction of acid radicles, notably acetyl and benzoyl, and of alkyl groups are fully described, and wherever chemicals rarely met with in laboratory practice are referred to, details for their preparation are given. The arrangement of the matter and the style of writing are all that could be desired, and the numerous references to original papers cannot but add to the utility of the book. The only typographical error noticed is on page 41, where the group carboxyl is represented as CHOH instead of COOH.

A. R. L.

DAIRY CHEMISTRY: A PRACTICAL HANDBOOK FOR DAIRY CHEMISTS, ETC. By H. D. RICHMOND, F.I.C. London: Charles Griffin and Co., Limited. Price 16s.

If the question were raised as to the necessity for a work specially treating on the chemistry of milk, it should certainly be answered in the affirmative. Not only is the public analyst called upon to examine large numbers of samples of milk and milk products with the object of detecting and checking adulteration, but other chemists are also frequently called upon to analyse and pass opinions on materials directly or indirectly connected with the dairy, and last, but not least, practical dairy work nowadays demands a considerable amount of scientific knowledge if it

is to be performed in a satisfactory and successful manner. The days are gone for ever in which a very limited amount of experience was considered either sufficient to judge whether a milk was pure or sophisticated, or to support and aid the practical dairyman with scientific counsel, or to fill up a responsible position in the dairy industry. If, then, a work on dairy chemistry must be considered highly desirable—nay, necessary—nobody could be better fit for producing such a work than the author of the book lying before us. Mr. Richmond has for nearly eight years superintended the Aylesbury Dairy Company's laboratory, which was erected in the year 1880. Although established to serve the special requirements of that large firm in exercising an extended and minute control over the milk and milk products there dealt with, Mr. Richmond, like his predecessor, has not confined himself to exercising control alone, but has taken good care to make extensive use of the opportunities given to work out as many questions as possible which in one way or another are connected with the chemistry of milk, milk products and dairy work generally. In the course of twenty years a vast amount of facts and experiences has been thus accumulated, which in itself would have been quite sufficient to form a valuable publication. But the author has done much more. By collating and studying the respective publications of others, and reproducing them in a digested form, he has succeeded in putting before the reader a complete work on dairy chemistry. It would take up far too much space merely to indicate the contents of the various chapters of the book, to draw attention to the useful tables, and to enumerate the numerous illustrations. Less still could justice be done to Mr. Richmond's work by an attempt to specially mention the most important parts and statements. In fairness we must also abstain from noticing the weaker portions of the book, and as these are scant and of little importance we can do so without remorse. Suffice it to say that Richmond's "Dairy Chemistry" forms a complete résumé of theoretical and practical knowledge, written in easily intelligible language, and is a work useful to everyone desiring instruction in the chemistry of milk and milk products, a work which the attentive reader will study with the greatest satisfaction. P. V.

A Text-book of Physical Chemistry. By Dr. R. A. Lehfeldt. London: Edward Arnold. Price 7s. 6d.

This branch of general chemistry has developed so enormously during the past twelve years or so that it now constitutes a department of considerable size and importance; and as its teachings already to some extent exert on analytical problems an influence which in all probability will become more marked as time rolls on, it behoves every analytical chemist to become acquainted with the general principles of this subject, especially as analytical processes depend ultimately on a knowledge, not only of chemistry in the restricted sense, but of the physical behaviour of the materials dealt with. The work before us is eminently adapted for this purpose; it forms a well-written digest, couched in terse but intelligible language. Although mathematical formulæ are indispensable in illustrating a subject of this nature, the work is ingeniously arranged so as to be intelligible to the non-mathematical reader who is content to take the mathematical proofs on trust. The general get-up of the book is good; it is printed in clear, bold type, contains numerous illustrations and has a copious index. W. J. S.

MISCELLANEOUS.

QUALIFICATIONS OF PUBLIC ANALYST.

THE following extracts are taken from an Order and accompanying Circular, dated March 7, 1900, issued by the Local Government Board to those local authorities who are required by law to appoint a Public Analyst:

"Every person appointed on or after the first day of January, one thousand nine hundred, to the office of Public Analyst shall furnish such proof as we may deem sufficient of his competent skill in, and knowledge of, (*a*) analytical chemistry, (*b*) therapeutics, and (*c*) microscopy.

"Such proof shall in every case comprise documentary evidence that such person holds the requisite certificate, diploma, license, or document conferring the qualification, or attesting his possession of the skill or knowledge to which the same applies, and granted or issued by any person or body of persons for the time being recognised by us as competent to confer such qualification, or to test such skill or knowledge. Such proof shall also comprise such further evidence as we may in any particular case require.

"All such documentary evidence as is hereinbefore mentioned shall be furnished by such person to the local authority by whom he is appointed, and shall be transmitted to us by the local authority when applying for our approval of the appointment.

"Provided that nothing in this regulation contained shall, in the case of any person who was appointed to the office of Public Analyst with our approval, between the first day of January, one thousand eight hundred and ninety-one, and the date hereof, or of any person who is so appointed for the first time after such last-mentioned date, apply upon any subsequent appointment of such person to the said office."

"As regards the reference in the Order to a person or body of persons whom the Board may from time to time recognise as competent to confer the requisite qualification, or to test the skill or knowledge of which proof is required by the Order, the Board may state that it would accord with their existing practice to accept as sufficient documentary evidence of the requisite qualification under the Acts the Diploma of Fellowship or Associateship of the Institute of Chemistry of Great Britain and Ireland, together with the Certificate granted by the Institute after an examination, conducted by them on lines approved by the Board, in therapeutics, pharmacology, and microscopy."

"The possession of a diploma as a registered medical practitioner is accepted as sufficient proof of competency in microscopy and therapeutics, and it would only be necessary that a medical practitioner appointed as a public analyst should furnish evidence of competent skill in, and knowledge of, analytical chemistry."

"Evidence of skill or knowledge on the part of a candidate in respect of any of the qualifications referred to as requisite, which is tendered by an individual, must be from a person recognised as entitled to speak with authority as to proficiency in the particular qualification in question."

THE ANALYST.

MAY, 1900.

PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS.

THE monthly meeting of the Society was held on Wednesday evening, April 4, in the Chemical Society's Rooms, Burlington House. The President, Mr. W. W. Fisher, M.A., occupied the chair.

The minutes of the previous meeting were read and confirmed.

Certificates of proposal for election to membership in favour of Messrs. T. E. Hill, M.B., B.Sc., F.I.C., T. Macara, and W. E. F. Powney were read for the second time; and certificates in favour of Messrs. John Richard Brooke, A.I.C., Analytical Chemist, 36, Mincing Lane, London, E.C.; Thomas Henry Lloyd, Analyst and Bacteriologist, The Laboratory, Penygraig, Rhondda; and George Harris Morris, Ph.D. (Würzburg), F.I.C., Analytical and Consulting Chemist, 72, Chancery Lane, London, W.C., were read for the first time.

Messrs. Arthur Alexander Ramsay, James Eugene Kirkpatrick, and A. J. Murphy were elected members of the Society.

The following papers were read: "Note on the Influence of Temperature and Concentration on the Saline Constituents of Boiler Waters," by Cecil H. Cribb, B.Sc.; "On an Improved Absorption Apparatus for Use in the Analysis of Essential Oils," by Alfred C. Chapman and Herbert E. Burgess; and "On the Composition of Danish Butters," by Harold Faber.

BUTTERS FROM VARIOUS COUNTRIES COMPARED.

BY C. ESTCOURT, F.I.C.

(*Read at the Meeting, February* 7, 1900.)

DURING the last twelve months I have analysed a considerable number of samples of butter for the direct importers of such produce, representing shipments from Ireland, Denmark, Sweden, Germany, Canada, and Finland.

I am able to give the following results of the analyses of about 250 samples, the origin of each of which I ascertained and noted:

Water in Butter.

Origin of Samples.	Total Samples.	Average Percentage of Water.	Highest Percentage of Water.	Lowest Percentage of Water.
Finnish	85	12·3	18·0	8·0
Danish	89	13·5	18·0	9·0
Irish	39	14·1	20·0	9·0
Swedish	14	13·75	17·8	11·8
Canadian	10	13·2	19·4	10·0
German	8	13·5	14·8	11·4

Water.

The percentages of water, as will be seen from the tables just given, vary considerably in the produce of every country with the exception of Germany.

I give in the foregoing tables the average, as well as the highest and lowest figures.

On reviewing the results, I find in Irish butter that, of the thirty-nine samples examined, only four contained more than 18 per cent. of water, three contained more than 16 but less than 18 per cent., as many as twenty-three contained from 12 to 16 per cent., and the results obtained from nine samples gave figures falling below 12 per cent.

In the case of the Danish samples only nine contained more than 15 per cent. of water, while six contained less than 12 per cent.

The produce from Finland showed that only three samples of the eighty-five analysed contained more than 15 per cent. of water, twenty-six contained between 13 and 15 per cent., and the remainder, fifty-six, were all under 13 per cent. The Finnish butter, therefore, must be deemed a most satisfactory article as regards the quantity of water.

Of the Swedish samples, six showed water to the extent of 14 per cent. and above, the highest being 17·8 per cent.

The Canadian samples gave results varying from 10 to 15 per cent. of water, with the exception of one sample which was found to contain as much as 19·2 per cent.

The German produce gave results much more constant: six of the eight samples analysed contained from 12 to 14 per cent., and one 14·8 per cent. of water. The exceptional one contained 11·4 per cent.

It will appear from the results obtained with the Irish butters that where care is exercised, as it is at the creameries, no great excess of water is found.

Samples Examined for Preservatives.

Origin of Samples.	Total Samples Examined.	Samples not containing Preservatives.	Samples containing Boric Acid.		
			Average in Grains, per pound.	Highest in Grains, per pound.	Lowest in Grains, per pound.
Finnish ...	74	73	8·3	—	—
Danish ...	85	82	5·3	8·0	4·0
Irish ...	37	2	24·7	46·0	5·0
Swedish ...	13	13	—	—	—
Canadian ...	10	9	9·0	—	—
German ...	8	8	—	—	—

Preservatives.

As regards the examination of the samples for preservatives, it will be seen from the tabulated results that not a single sample of the German and Swedish imports was found to contain any.

Of the ten Canadian butters, one contained boric acid, and that only to the extent of 9 grains per pound.

Eighty-five specimens of Danish butter were examined, and in three samples only could any preservative be found, namely, boric acid, and the highest quantity contained in a sample was 8 grains per pound.

Of the seventy-four samples from Finland, only one was found upon examination to contain boric acid, namely, 8·3 grains per pound.

Therefore the percentage of the samples of Danish and Finnish butter containing preservative is very low and scarcely worthy of notice.

The same cannot be said of the Irish production, of which all the samples were found to contain boric acid, varying in quantities from 5 to 46 grains per pound.

Of the thirty-five samples in which preservative was found, eight contained upwards of 35 grains per pound of boric acid, including two containing above 40 grains.

Nineteen showed results between 20 and 35 grains per pound, and seven samples contained quantities below 12 grains of boric acid.

These results are rather surprising following upon those of the analyses of the produce of other countries, which rather tend to show that a preservative is not absolutely necessary.

Figures obtained by the Reichert Process.

Origin of Samples.	Total Samples.	Average Figure.	Highest Figure.	Lowest Figure.
Finnish	85	13·8	17·6	11·9
Danish	90	15·2	17·0	12·5
Irish	39	14·0	15·5	12·8
Swedish	14	14·4	16·2	13·7
Canadian ...	10	14·1	15·5	13·0
German	8	15·3	16·2	13·5

Results obtained by the Reichert Process.

Coming to the composition of the fats of these different butters, the results obtained by submitting them to the Reichert test are given below; and a few notes I have made may be of some interest to the members of this Society.

Taking the Finnish butters first, the fats of eighty-four samples were examined, and gave an average Reichert figure of 13·8; only eight gave Reichert figures of 16 and over; whilst twenty-one samples showed results below 13, including two results of 11·9.

These two samples I returned as adulterated, and they accordingly were sold as confectionery butter.

With regard to the Irish butters, only four gave Reichert numbers below 13, and

seventeen samples were lower than 14; there was not much variation among the remainder of these thirty-nine samples, the figures of which ranged from 14 to 15·5.

Of the Danish samples, ninety were analysed, and as many as twenty-two gave Reichert figures of 16 and above, including two of 17, showing a very large percentage having a high Reichert number. There were only three giving numbers of 13 and lower, the general figure for the remaining sixty-five samples being about 15.

Denmark may be said to produce a butter of very good quality, and the Reichert figure of which is fairly constant.

The butters of German and Swedish origin appear to be of even composition, as evidenced by the results obtained by the Reichert test.

Of the Swedish, only one of the fourteen samples gave a figure so low as 13·7, the next lowest being 14·4, and this is the average figure for the whole of these samples.

The results of the German butters were higher on the whole than the Swedish, though the lowest in this case was 13·5.

Although the Canadian and Irish agree practically as regards the average Reichert figure, the former have a greater percentage of samples whose figures come as low as 13 to 13·5. Four out of the ten are below 13·5, while the remainder agree fairly with the Irish.

NOTES ON SOUR MILK.

By H. Droop Richmond, F.I.C., and J. Bristowe P. Harrison, A.I.C.

(*Read at the Meeting, February* 7, 1900.)

I. The Determination of the Specific Gravity of Sour Milk.

Weibull (*Chem. Zeit.*, 1893, xvii. 1679) proposed, in order to determine the specific gravity of sour milk, the addition of a known volume of ammonia; the density of the mixture, corrected for the volume of ammonia added, of which the density is known, gives the density of the milk.

It has been our practice to slightly modify this method; we add to each 100 c.c. of sour milk 5 c.c. of strong ammonia, and to the density of the mixture we apply a constant correction deduced from the change of density on adding 5 c.c. of strong ammonia to 100 c.c. of fresh milk; this correction has varied from 0·0065 to 0·0070 with different samples of strong ammonia.

We can fully confirm Weibull's statement that the density of sour milk can be accurately determined by this means, and have used the method extensively.

De Koningh (Analyst, xxiv. 142) has substituted a solution of caustic soda (specific gravity 1·030) for the strong ammonia; he finds that a constant correction of 0·0008 must be added to the results, this being the mean figure by which the density is lowered on mixing 5 c.c. of soda solution with 100 c.c. of milk. He accounts for the lowering of density by the assumption that calcium phosphate is rendered insoluble; we neither entirely confirm his figure nor are we satisfied with his explanation; as we propose to show, the correction is not constant, but varies

with the "acidity" of the milk, and can be, we think, explained when the action of acids on alkalies is considered.

In Table I. we give the densities of solutions of caustic soda, ammonia, and sulphuric and hydrochloric acids, and the salts resulting from their mutual action.

The figures are obtained by interpolation from well-known tables, which we have taken from the *Chemiker Kalender*, and are expressed in terms of $\frac{N}{5}$, $\frac{N}{10}$, and $\frac{N}{20}$ solutions; they are probably correct to the fourth place of decimals.

TABLE I.

Densities of Solutions of Bases, Acids, and Salts.

Substance.	$\frac{N}{5}$.	$\frac{N}{10}$.	$\frac{N}{20}$.	Authority
NaOH ...	1·00920	1·00465	1·00235	Lunge.
NH_3 ...	0·99856	0·99928	0·99964	Carius.
HCl ...	1·00372	1·00186	1·00093	Lunge and Marchlewski.
H_2SO_4 ...	1·00774	1·00387	1·00193	Lunge and Isler.
NaCl ...	1·00846	1·00423	1·00212	Gerlach.
NH_4Cl ...	1·00338	1·00169	1·00085	,,
Na_2SO_4 ...	1·01266	1·00639	1·00322	,,
$(NH_4)_2SO_4$	1·00752	1·00376	1·00188	Schiff.

If a salt in solution had a density which was the sum of the densities of the acid and base of which it was composed, the density of a solution of a salt should be equal to the sum of the densities of corresponding strengths of its acid and its base, less 1. This is not, however, the case, and the difference between the sum of the densities of the acid and base (less 1), and the density of the salt is given in Table II.:

	NaOH.		NH_3.	
	HCl.	H_2SO_4.	HCl.	H_2SO_4.
$\frac{N}{5}$ ...	−0·00446	−0·00428	+0·00110	+0·00122
$\frac{N}{10}$ ...	−0·00228	−0·00213	+0·00055	+0·00061
$\frac{N}{20}$ ...	−0·00116	−0·00106	+0·00028	+0·00031

As the acidity of fresh milk is about $\frac{N}{50}$, and that of sour milk about $\frac{N}{20}$ to $\frac{N}{10}$, it is seen that the change of density on neutralization will go a long way towards explaining the change noticed by De Koningh.

Thinking that it would not be quite correct to compare the acids such as sulphuric and hydrochloric, with milk, we have made a few experiments with weaker acids.

We prepared a solution of caustic soda of density 1·032, which was 0·666 N., and determined the densities of various dilutions of this with water. There were:

No. of c.c. diluted to 100 c.c. $= x$.	Density found.	Density calculated.
4·10	1·00138	1·00138
7·96	1·00266	1·00265
11·81	1·00388	1·00391

Formula: Density $= 1 + 0{\cdot}000339x - 0{\cdot}0000007x^2$.

The determinations show that when 5 to 20 c.c. of this solution are added to 100 c.c. the density is about 0·0001 higher than that calculated proportionally.

A solution of citric acid, 1·0064 N., was prepared; the densities of various dilutions were determined:

No. of c.c. diluted to 100 c.c. = x.	Density found.	Density calculated.
2·39	1·00059	1·00064
4·80	1·00135	1·00130
7·47	1·00200	1·00201

Formula: Density = $1 + 0{\cdot}0002688x$.

Solutions were now made up containing varying quantities of citric acid and a slight excess of soda solution:

No. of c.c. of Citric Acid in 100 c.c.	No. of c.c. of Soda in 100 c.c.	Density found.	Density calculated.
2·41	4·04	1·00174	1·00200
4·82	8·48	1·00361	1·00412
7·41	11·99	1·00524	1·00595

The calculated density was obtained by adding together the figures obtained by calculating the densities due to the citric acid and soda by the formulæ given above, and subtracting 1.

Fifty c.c. of a solution of lactic acid diluted to 100 c.c. (making 0·0256N) had a density of 1·00060; 50 c.c. of this solution + 4·76 c.c. solution of caustic soda, diluted to 100 c.c., had a density of 1·00195. The calculated density was 1·00220.

Calculated to a strength of $\frac{N}{10}$, the differences are for citric acid 0·00105, 0·00104, and 0·00095, or a mean of 0·00100, and for lactic acid 0·00098, figures which are less than half that given by the stronger acids. As citric acid, in addition to being a weak acid, is a tribasic acid, we thought that the lower figure obtained might be partly due to the three hydrogen atoms not having the same value. It was convenient to test this question in the first instance with oxalic acid on account of the ease with which normal and acid salts could be prepared.

We made a quantity of each salt, the purity of which is shown by the following figures:

	Normal Salt $Na_2C_2O_4$		Acid Salt $NaHC_2O_4{\cdot}OH_2$	
	Found	Theory	Found	Theory
Total C_2O_4	65·55, 65·48	65·67	67·58, 67·82	67·69
C_2O_4 (as acid)	none	none	33·61	33·85
Loss in water-bath	none	none	13·92	13·85
Further loss at 105° to 110°	none	none	none	none

The following densities were determined:

(i.)	Oxalic acid 0·0406 N.	1·00082
	,, ,, 0·10 N.	1·00217
	,, ,, 0·2597 N.	1·00563
(ii.)	*$Na_2C_2O_4$ 0·10 N.	1·00556
(iii.)	*$NaHC_2O_4$ 0·20 N.	1·00720
(iv.)	Oxalic acid 0·10 N., to which 14·43 c.c. soda solution per 100 c.c. had been added = 0·0961 N.	1·00572

* These two solutions contained the same amount of sodium.

From the above figures we calculate the following losses of density for $\frac{N}{10}$ solutions:

When the total acidity is neutralized	from i., ii.,	and density of NaOH	0·00126
,, ,, ,, ,,	from i. and iv.	... ...	0·00124
When the first hydrogen atom is neutralized	from i., iii.,	and density of NaOH	0·00179
When the second hydrogen atom is neutralized	from i., ii., and iii.	... ...	0·00053
,, ,, ,, ,, ,,	from i., iii., and iv.	... ...	0·00068
,, ,, ,, ,, ,,	from ii., iii.,	and density of NaOH	0·00073
,, ,, ,, ,, ,,	from i., ii., iii., and iv.	... ...	0·00071

From the above results it appears that the first hydrogen atom in oxalic acid is the more powerful.

It is seen from the above that although neutralization of an acid by soda always produces a loss of density, the figure varies not only with the acid, but also with the hydrogen atom neutralized in a polybasic acid. For this reason it is useless to apply any theoretical correction for milk, and we have made some experiments to see if we can find a value which may be used in milks.

To milk of density 1·0319 increasing quantities of soda solution were added and the densities taken. Three c.c. of the solution rendered this milk exactly neutral to phenolphthalein.

Quantity added.	Density.
1 c.c. to 100 c.c.	1·03175
2 ,, ,,	1·0316
3 ,, ,,	1·0316
4 ,, ,,	1·0316

Five c.c. of soda solution was added to 100 c.c. of different milks, and the densities determined.

Original ...	1·0325	1·0327	1·0319	1·0328	1·0339 1·0338	1·0309	1·0328
Alkaline ...	1·0322	1·0321	1·0316	1·0324	1·0334 1·0335	1·0306	1·0325

The third milk in the above table was allowed to get sour; when the acidity was equal to 9 c.c. of the soda solution for 100 c.c., it still was liquid, and had a density of 1·0319. On the addition of 5 c.c. soda solution the density fell to 1·0316, and to 1·0310 by the addition of 10 c.c.; when 15·1 c.c. were required to neutralize it, the addition of 20 c.c. of soda dissolved the precipitated curd, and the density was 1·0298. In each case Weibull's method, applying the correction found for fresh milk, gave a density of 1·0320.

The following additional experiments were performed:

Original Density of Milk.	Acidity.	Soda added.	Density of Alkaline Milk.
1·0331	136°	21 c.c.	1·0300
1·0326	124°	19 c.c.	1·0302

Fresh milk has, according to our experience, an average acidity of 20°, and we assumed that all the milks tested in a fresh condition had this acidity.

Tabulating the acidities and the lowering of density, we get:

Original Density.	Acidity Neutralized.	Lowering of Density.	Calculated Original Density.
1·0319	6·7°	0·00015	1·0319
,,	13·3°	0·0003	1·0319
,,	20·0°	0·0003	1·0320
1·0325	20·0°	0·0003	1·0326
1·0327	20·0°	0·0006	1·0325
1·0319	20·0°	0·0003	1·0320
1·0328	20·0°	0·0004	1·0328
1·03385	20·0°	0·0004	1·03385
1·0309	20·0°	0·0003	1·0310
1·0328	20·0°	0·0003	1·0329
1·0319	33·3°	0·0003	1·0323
,,	60·0°	0·0009	1·0321
,,	101·0°	0·0021	1·0318
1·0326	124·0°	0·0024	1·0327
1·0331	136·0°	0·0031	1·0327

The figures calculated are obtained on the assumption that each degree of acidity causes a lowering of the density of 0·00002, and with this correction the results are fairly accurate.

On referring to Table II. we see that with strong acids the change of density on neutralizing with ammonia is very much smaller than with soda, and in the opposite direction, and our results with milk indicate that it may practically be neglected. This is due partly to the fact that dilution of strong ammonia produces a small change in density in the direction opposite to that due to neutralization, and partly to the fact that there may be a small loss in solids when milk becomes sour, and the small errors in the one case tend to neutralize the small errors in the other.

With De Koningh's method the errors are larger, and tend in the same direction, and though an approximate correction depending on the acidity of the milk can be worked out, it appears preferable to use Weibull's method.

II. The Point at which Milk may be considered Sour, and the Rate of Souring in the Presence or Absence of Preservatives.

There is considerable divergence of opinion as to the degree of acidity at which milk may be considered sour. Thus Stokes (Analyst, xvi., 122) states that milk which has not yet reached an acidity of 0·3 per cent. lactic acid (33·3°), but is near it, will coagulate on boiling; he records, however, the fact that three samples did not coagulate when the acidity had reached 0·54 per cent. lactic acid (60°). He also gives figures which show that milk tastes sour at an average acidity of 0·396 per cent. lactic acid (44°). Thorner (*Chem. Zeit.*, 1891, 1108) gives 23° as the acidity at which milk curdles on boiling, and gives the normal acidity of milk as 12° to 16°.

Rideal and Foulerton (*Public Health*, May, 1899) say that milk turns sour at an acidity of 0·5 per cent. lactic acid (55·6°), but Rideal (*Lancet*, January 27, 1900, 228) finds that milk curdles on boiling at 28° acidity, while with other samples slight coagulation was noticed at 25·6° and 21·6°.

We have made a series of experiments, and our figures confirm almost absolutely

the figures of Stokes. On the average we find that milk tastes sour at 45°, though the variations are fairly wide. The following experiments show the point at which milk curdles on boiling:

	Acidity.	Remarks.
i.	33·3°	Faintly curdles on boiling.
ii.	28·0°	Does not curdle on boiling.
iii.	52·0°	Curdles on warming.
iv.	32·7°	Curdles on boiling.
v.	33·3°	Faintly curdles on boiling.
	34·0°	Curdles on boiling.
vi.	24·7°	Does not curdle on boiling.
vii.	37·8°	Curdles on boiling.
viii.	34·9°	Just curdles on boiling.
ix.	32·5°	Just curdles on boiling.

The acidity at which milk curdles on boiling is about 33°, and as we find that milk, when fresh, has an acidity of 20°, this corresponds to an increase of 13°. We have tried to ascertain the amount of acid added to milk which will curdle it on boiling; aqueous solutions of various acids were added little by little with constant shaking, till a faint coagulation on boiling was observed. The results were:

Acid.	Volume necessary expressed as $\frac{N}{10}$ c.c. to 100 c.c.
Sulphuric	8·8
Hydrochloric	8·6
Oxalic	29 and 28
Lactic	9·7

We find that milk curdles at about 85°, or an increase of 65°, without boiling; this was the figure found at 17° to 35°, and between these limits does not appear to vary appreciably; it is difficult to determine the point with accuracy.

It is quite certain that the "acidity" of milk is not wholly due to lactic acid; indeed, the "acidity" of fresh milk is due to the mono- and di-basic phosphates, and not to free acid at all. Seeing that 9·7 c.c. $\frac{N}{10}$ lactic acid will curdle milk on boiling, while it requires a development of about 13° "acidity," it is highly probable that another acid very much weaker than lactic is produced, and we venture to think that carbonic acid is responsible for a portion of the acidity of sour milk; we know that carbonic acid is produced, and we have found that when milk is sufficiently sour to develop gas about half the acidity, as indicated by phenolphthalein, is shown to litmus (to which both milk and carbonic acid are approximately neutral).

We have based a hypothesis on the facts that different acids do not give the same result, that salts of polybasic acids are present in milk, that both casein and albumin have acidic functions, and that the coagulation of milk at temperatures between 17° and 35° does not appreciably vary with the temperature; it appears to us that curdling of milk is due to an amount of acid being present to set up an equilibrium between the acids and bases present, such that certain acids, *e.g.*, casein and albumin, are liberated. At a boiling temperature we are inclined to think that the curdling is determined by the coagulation of the albumin, the equilibrium being destroyed by the removal of one acid (albumin) from solution, and fresh amounts of albumin, and finally perhaps casein, are liberated.

When milk tastes sour, it would appear that the equilibrium is such that a sour-tasting free acid exists in solution; while when milk curdles spontaneously the equilibrium is such that the insoluble acid casein is produced.

We have mentioned this hypothesis to emphasize the fact that the point at which milk must be considered sour is a purely arbitrary one; the three points we have mentioned are determined by the coagulation of albumin, the production of a sour-tasting acid, and the formation of insoluble casein respectively, and others could doubtless be found.

We have made some experiments to determine the rate of souring of milks with and without preservatives. The preservatives used were a mixture of borax and boric acid, in the proportion of one part borax to three parts boric acid, from which sufficient water had been driven off to raise the percentage of B_2O_3 to 56·3 per cent. (this is a mixture largely sold under fancy names), and a solution of formaldehyde.

Our results are, with boric preservative (the figures represent degrees of acidity):

Temp. 17·5° C.	Milk alone.	Milk + 0·05 per Cent.	Milk + 0·10 per Cent.
52 hours	69·9°	31·3°	35·3°
76 ,,	95·0°	67·3°	52·6°
Temp. 24·5° C.			
24 hours	32·6°	31·3°	35·0°
48 ,,	99·2°	97·1°	82·6°
Temp. 34·5° C.			
22·5 hours	93·6°	71·3°	45·3°
46 ,,	116·6°	114·8°	108·2°

The acidity of the milk when fresh was:

Milk alone	19·7°
,, + 0·05 per cent.	25·3°
,, + 0·10 ,, ...	31·3°

With formaldehyde:

Temp. 17·5° C.	Milk alone.	Milk + 0·0023 per Cent.	Milk + 0·0047 per Cent.	Milk + 0·0093 per Cent.
24 hours	22·9°	—	—	—
48 ,,	72·7°	29·3°	23·3°	22·0°
72 ,,	93·6°	99·2°	33·3°*	24·0°
Temp. 27° C.				
24 hours	58·7°	26·7°	23·3°	—
48 ,,	106·3°	115·3°	81·3°*	27·3°
72 ,,	118·0°	120·8°	123·3°	56·7°
Temp. 36° C.				
24 hours	83·3°	80·0°	28·0°	22·7°
48 ,,	116·7°	141·6°	129·2°	52·0°*
72 ,,	158·3°	152·4°	139·2°	132·0°

The acidity of the fresh milk was in all cases 20·7°.

In the samples marked with an asterisk we could only obtain a very slight formaldehyde reaction with Hehner's tests. All the samples with an acidity below 30° gave a strong reaction, and the others a most indefinite one.

We would draw attention to the fact that when a milk treated with formaldehyde

begins to develop acidity, the rate of souring is greater than that of untreated milk, and this is noticed also, to a smaller extent, with boric preservative.

By drawing a curve including the results at 17·5°, we get an expression of the rate of souring, and we find that the same curve with a different time factor applies without great error to the other temperatures, and to the results with boric preservative; a second slightly differing curve applies to the formaldehyde results.

By means of these curves we have constructed tables of the time which milk takes to arrive at the three points of "sourness"—*i.e.*, an increase of 13°, 25°, and 65° acidity. These tables are, of course, only approximate, but we do not think that the deviation exceeds 10 per cent. of the values stated.

Table of the Time in Hours taken by Milks to reach 13° Additional Acidity.

		Boric Preservative.		Formaldehyde.		
Temp.° F.	Milk alone.	Milk + 0·05 per Cent.	Milk + 0·10 per Cent.	Milk + 0·0023 per Cent.	Milk + 0·0047 per Cent.	Milk + 0·0093 per Cent.
60	42	71	93	51	85	119
70	29	36	46	34	49	78
80	19	22	31	25	34	56
90	13	15	23	15	26	44
100	7·5	10	20	9	23	37

Time in Hours to reach 25° Additional Acidity.

		Boric Preservative.		Formaldehyde.		
Temp.° F.	Milk alone.	Milk + 0·05 per Cent.	Milk + 0·10 per Cent.	Milk + 0·0023 per Cent.	Milk + 0·0047 per Cent.	Milk + 0·3093 per Cent.
60	50	84	110	60	100	140
70	34	43	54	40	58	92
80	22	26	36	29	40	66
90	15	18	27	18	31	52
100	9	12	23	11	27	44

Time in Hours to reach 65° Additional Acidity.

		Boric Preservative.		Formaldehyde.		
Temp.° F.	Milk alone.	Milk + 0·05 per Cent.	Milk + 0·10 per Cent.	Milk + 0·0023 per Cent.	Milk + 0·0047 per Cent.	Milk + 0·0093 per Cent.
60	75	126	165	84	140	192
70	51	64	81	56	81	129
80	33	39	54	41	56	92
90	22	27	40	25	43	73
100	12·5	18	34	15	38	62

The only results we have been able to find which are comparable with ours are those of Rideal (*loc. cit.*). Applying the same methods to his results, we obtain practically the same figures as those given above.*

We would draw attention to the fact that at high temperatures (say 80° hot summer weather) preservatives are comparatively useless unless added in relatively

* We assume that as Rideal does not notice the increased acidity when boric acid is added to milk, he has subtracted a constant amount for this throughout his series.

large quantities; the minimum quantities used by us, and also by Rideal, only increase the life of milk a few hours, and are equivalent only to a lowering of temperature of about 5° F. Unless milk can be made to keep at least twelve hours longer than without preservatives, we do not think much is gained by their use, and to do this in summer we think that the minimum amounts are 0·09 per cent. boric preservative and 0·004 per cent. formaldehyde.

We would also draw attention to the increased rate of souring as time goes on, when preservatives are added. This indicates a possible danger in using preservatives in milk, as it seems far from improbable that succeeding generations of micro-organisms become in the presence of preservatives more active and more virulent, and if the use of preservatives were universal, there is a probability that they would cease to act.

The work of Effront on yeast grown in the presence of sodium fluoride shows that this view is not a mere hypothesis.

Discussion.

The Chairman (Mr. Allen) inquired whether there was any reason to suppose that milk contained carbonic acid or carbonates. If this were the case, the reliability of titrations with phenol-phthalein would be seriously affected.

Mr. Richmond said that a portion of the acidity of sour milk was undoubtedly due to carbonic acid, and the question had been considered in connection with this paper. The figures, however, were not given as representing the acidity absolutely, but were only intended for comparative purposes. The question of carbonic acid would not affect the immediate object of the determinations, which was to ascertain the time required for the acidity to reach a certain point. If different samples contained different proportions of carbonic acid, there would, of course, be a distinct error; but these samples had been treated as nearly as possible under the same conditions, and the ratio of carbonic acid to other acids was probably not sufficiently divergent to affect the conclusions.

Mr. Chapman asked whether the experiments took place in the winter or summer months. The effect of a temperature of 80° F. in the summer and in the winter would probably not be the same.

Mr. Richmond said that the experiments were conducted during the winter. If any difference occurred, it would be rather in the autumn months—September and October—when an increase in the rate of souring might be expected. He did not think there would be any increase in the summer.

ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

ORGANIC ANALYSIS.

The Detection of Cotton-seed and Sesame Oils in Fats. J. Wauters. *Bull. de l'Ass. belge*, 1899, xiii., 404-416.)—From the results of his experiments on the detection of these oils in butter, lard and other oils, the author has arrived at the following conclusions:

Halphen's reagent is preferable to Becchi's reagent for the detection of cotton-seed oil. It does not produce any coloration with other oils or fats, and is capable of detecting 0·25 per cent. of cotton-seed oil in mixtures. The reaction, which is also given by old oils, gains in sensibility by decolorizing the fat by heating it at about 50° C. with animal charcoal. Butter, both naturally and artificially coloured, and margarine are completely decolorized in this way.

The fatty acids do not give the coloration after being dried at 100° C., but with the undried acids the reaction is obtained with even greater intensity than with the fat itself.

Butters prepared from the milk of cows partially fed on cotton-seed cake give the reaction, but the intensity of the colour does not appear to exceed that produced by a fat containing 1 per cent. of the oil. On the other hand, feeding the cow with cotton-seed cake does not appear to have any influence on the chemical or physical constants of the butter. Decolorization by means of animal charcoal does not remove from sesame oil the active principle which gives the coloration in the Baudouin test. It removes foreign colouring matters, however, and notably turmeric. By applying the test to the decolorized fat it is possible to detect as little as 0·25 per cent. of sesame oil in butter or margarine. C. A. M

Notes on the Constitution of Train Oils. H. Bull. (*Chem. Zeit.*, 1899, xxiii., 996.)—By suitable treatment with alkalies dissolved in absolute alcohol, the fatty acids in train oils can be divided into four groups: (*a*) those whose potassium salts crystallize out of alcohol, (*b*) those whose sodium salts are crystallizable, (*c*) those whose sodium salts are very soluble in ether, and (*d*) a small residue. From 1 kilo. of Baltic cod oil the fractions were as follows:

	Weight in Grammes.	Acid Number.	Iodine Number.
A	334	194·2	67·5
B	375	190	135·6
C	120	167	322·4
D	69	169	347

A contains practically all the saturated acids and some belonging to the oleic series; B contains acids of the oleic and linolic series; C consists chiefly of acids of the

formulæ $C_nH_{2n-8}O_2$ and $C_nH_{2n-10}O_2$ ($C_{20}H_{32}O_2$ and ? $C_{23}H_{30}O_2$). For the latter an iodine value of 369 and an acid value of 165·2 were found, which agree satisfactorily with theory.

An imperfect separation of the fatty acids of the original oil can also be attained by distilling them at 200° C. in a sharp current of steam heated to the same temperature, recovering the distillates by fractional condensation in a set of receivers maintained at 180°, 160°, 140°, 120°, and 100°. The same process may also be applied to the acids previously separated by crystallization; fraction A above gave the following results:

Temperature of Receiver.	Weight of Fraction.	Acid Number.	Iodine Number.
160°	83·3	182	102·6
140°	95·5	200	62·4
120°	115·7	209	40
100°	14·2	212	33

Saturated acids were present in all fractions: stearic acid only in the first; stearic acid with a little palmitic in the second; palmitic almost alone of the saturated acids in the third; in the last palmitic and its lower homologues.

The ordinary processes for separating fatty acids which depend on the use of lead are not suited for the investigation of train oils. In all the samples examined, except torsk,* erucic acid, $C_{22}H_{42}O_2$, was met with, and also a new acid, $C_{20}H_{38}O_2$, melting about 20° — both of which (especially the former) yield lead salts slightly soluble in ether. In liver oil a new acid of the oleic series containing 21 atoms of carbon was found, which melted at 24·5°, and had the acid value 171·6 (theory, 172·8); this gave a lead salt very soluble in ether, and was therefore no mere mixture of the two former. In herring oil two unsaturated liquid acids with melting-points below – 20° were discovered, which gave the annexed analytical results:

Formula.	Acid Value.	Iodine Value.
$C_{20}H_{32}O_2$	179·0	344·5
$C_{24}H_{40}O_2$	154·9	279

F. H. L.

Estimation of Unsaturated Fatty Acids in Train Oils. H. Bull. (*Chem. Zeit.*, 1899, xxiii., 1043.)—This process depends, as already mentioned (*cf.* preceding abstract), on the solubility in ether of the sodium salts of the unsaturated acids of train oil. Seven grammes of the oil are weighed into a 200 c.c. flask, mixed with 25 c.c. of sodium ethylate (23 grammes of metal dissolved in 1 litre of absolute alcohol), and boiled on the water-bath for half an hour under an inverted condenser, shaking at intervals. The flask is closed and set aside for two hours; the soaps are thoroughly broken up with a rod, and 144·2 c.c. of ether free from alcohol and water (which has stood at least twenty-four hours over a large quantity of calcium chloride) is introduced. After agitating several times at intervals of half an hour, the mass is thrown on a dry 150 millimetre filter, the funnel of which is covered with a plate moistened with glycerin, and is connected by a rubber cork

* A fish allied to the cod.

to the flask, a piece of string being inserted between paper and funnel to permit circulation of air. One hundred c.c. of the filtrate (equivalent to 4 grammes of oil) are removed and extracted three times with 20 c.c. of water, a little free alkali being added each time in presence of phenolphthalein to prevent dissociation of the soaps. Any emulsion is broken down by means of a trace of alcohol or a few drops of alcoholic potash. The fatty acids are then liberated from the soaps, and the unsaponifiable matter extracted.

A very long table is appended giving the various constants of different varieties of train oil. Excepting the Japanese kinds, torsk oils, especially if pale, show a high proportion (12 to 21 per cent.) of unsaturated acids belonging to the series $C_nH_{2n-8}O_2$. The whale oils of the Northern Seas contain much less (4 to 9 per cent.); the antarctic "right" whale oil gives 19·5 per cent. Sardine oils contain 14 to 26 per cent. A turbid Japanese sample, however, gave only 5·75 per cent. The figures yielded by American porpoise-jaw oil show that an almost saturated acid of low molecular weight in it has a soluble sodium salt. Treated similarly, linseed, olive and rape oil give 2·37, 1·32, and 1·35 per cent. of soluble sodium salts respectively. F. H. L.

Estimation of Sugar in Transparent Soaps. F. Freyer. (*Oesterr. Chem. Zeit.*, 1900, iii., 25.)—The expression "glycerin soap" is used in trade to designate all transparent soaps, whether they contain glycerin or not. Materials intended for exportation to hot climates are nearly always prepared with cane-sugar, instead of glycerin, as the former enables them to bear high temperatures without sweating; and the proportion of sugar usually lies between 10 and 15 per cent. 16·28 grammes (a quarter of the normal weight per 250 c.c. for the soleil-ventzke-scheibler polarimeter) are dissolved on the water-bath in 50 or 100 c.c. of water, and a small excess (about 40 c.c.) of 10 per cent. barium chloride solution is added. The mixture is brought into a graduated flask, and diluted to 260 c.c., allowing 10 c.c. for the volume of the precipitated soap. The filtrate is tested with Fehling's solution for reducing sugars, polarized in the S-V-S instrument as it is, and polarized again after inversion with acid. F. H. L.

The Determination of Tannin and Gallic Acid. F. Jean. (*Rev. de Chim. Ind.*, 1900, xi., 35-38.)—This is an iodometric method based upon the fact that iodine combines with tannin or gallic acid to form compounds which do not give a blue coloration with starch. The iodine is dissolved in a solution of potassium iodide, and standardized on 0·1 per cent. solutions of tannin and gallic acid dried at 100° C. In each case 20 c.c. of the 0·1 per cent. solution are mixed with 5 c.c. of a concentrated solution of sodium bicarbonate, and the iodine run in drop by drop with continual circular agitation until a drop of the liquid gives a blue coloration when applied to paper covered with powdered starch.

The iodine solution is then strengthened or diluted according to the results of the titration until 10 to 10·5 c.c. are equivalent to 10 c.c. of the tannin solution. Gallic acid requires about 3 c.c. more of the iodine solution than tannin.

A blank determination is made to obtain the correction (usually about 0·4 c.c.) for the sodium bicarbonate.

In extracting the tannin substances from wood, leaves, etc., about 1 gramme of the sample is finely powdered, and heated for about thirty minutes with 15 c.c. of water at 50° C. The extract is decanted, the residue boiled for ten minutes with a fresh portion of water, and the aqueous solution again decanted, this treatment being repeated until the total extracts amount to nearly 100 c.c., to which volume it is finally made up when cold.

In the case of liquid extracts a solution is prepared containing 0·5 per cent. of the sample.

Ten c.c. of the solution are titrated with the standard iodine solution in the manner described above, the titration being repeated until the amount corresponding with 10 c.c. of the iodine solution is found. From the result the total amount of astringent matter is calculated.

For the separation of the tannin a solution of albumin is employed. This is prepared by triturating 2 grammes of dried egg albumin with sufficient glycerin (28-30° Ré.) to form a thin paste, leaving this for thirty minutes, and then making up to a litre with tepid water. It is preserved by the addition of a few fragments of camphor and by closing the flask with cotton wool impregnated with formaldehyde.

In the analysis 50 c.c. of the astringent solution are mixed with 15 c.c. of the albumin solution and 20 grammes of powdered sodium chloride, and the liquid made up to 100 c.c. with water, well shaken and filtered, the first portions being rejected.

A quantity of the filtrate, double that required in the first titration, is rendered acid by the addition of one drop of acetic acid and boiled to coagulate the excess of albumin. The filtrate and washings from this precipitate are cooled, mixed with 5 c.c. of sodium bicarbonate, and titrated with the standard iodine solution, a correction (usually about 0·7 c.c.) being made for the albumin from the result of a blank titration.

The amount of gallic acid is calculated from the iodine consumed, and the tannin is obtained from the difference in the results of the two titrations.

When the tannin material contains amylaceous substances soluble in water, the extraction should be made with alcohol. In the case of liquid extracts the absence of sulphites should be ascertained. C. A. M.

Detection of Nicotine. J. Schindelmeiser. (*Pharm. C. H.*, 1899, xl., 703; through *Chem. Zeit. Rep.*, 1899, 361.)—If unresinified nicotine is mixed with a drop of 30 per cent. formaldehyde, and a drop of strong sulphuric acid is added, an intense rose-red colour is produced, which becomes dark red in the presence of 0·005 or 0·01 gramme of the alkaloid. Concentrated formic acid may equally replace the formalin. F. H. L.

The Determination of Phosphorus in Organic Compounds. C. Marie. (*Bull. Soc. Chim.*, 1900, xxiii., 44, 45.)—One gramme of the substance is dissolved in 15 to 20 c.c. of concentrated nitric acid, the liquid gently heated, and finely divided potassium permanganate added in successive small portions until the colour of the solution remains pink for some minutes after the last addition. In the case of com-

pounds of the fatty acid series the destruction of the organic matter is very rapid; but with aromatic compounds it is more tedious, and it is necessary to add the permanganate in very small portions at a time, and to wait until the reaction is completed before adding the next portion.

A 10 per cent. solution of an alkali nitrite is added drop by drop to the oxidized liquid when cold until it becomes quite clear. It is then boiled for some time to expel the nitrous gases and to concentrate the solution, and the phosphoric acid precipitated in the usual manner with molybdate solution, care being taken to wash the precipitate completely free from manganese.

The author has tested this method on a large number of compounds, including some which, like Baeyer's ammonium calcium aceto-diphosphite, are extremely difficult to oxidize, even with fuming nitric acid in a sealed tube at 200° C., and has obtained results absolutely identical with those yielded by the ordinary method.

By the use of this method the analysis of glycero-phosphates may be greatly simplified. C. A. M.

A Simple Method of Estimating Mercury in Urine. Schumacher and W. L. Jung. (*Zeit. anal. Chem.*, 1900, xxxix., 12-17.)—A litre of the urine under examination is mixed with 15 to 20 grammes of potassium chlorate and about 100 c.c. of concentrated hydrochloric acid, and heated in a 2-litre flask on the water-bath until chlorine commences to be evolved. The flask is then allowed to stand for twelve hours. After expelling the excess of chlorine by gentle heating, the mercury is precipitated by the addition of 100 c.c. of a clear solution of stannous chloride, and the liquid filtered through purified asbestos. The precipitate, which contains a little organic matter, is washed by means of water and potassium hydroxide solution into a 300 c.c. flask, and gently heated so as to dissolve the organic matter; the solution is again treated with potassium chlorate and hydrochloric acid.

After filtration, the solution, while still warm, is treated with 10 to 20 c.c. of a concentrated solution of stannous chloride, and filtered through a Soxhlet-tube containing a layer of asbestos, and above it asbestos impregnated with metallic gold, which retains the mercury.

The asbestos is washed with dilute hydrochloric acid and water, then with alcohol and with ether, and the tube dried to constant weight in a current of dry air. The mercury is then expelled from the asbestos by heating the tube strongly and conducting a current of air through it until the weight again becomes constant. The difference between the two weights gives the amount of mercury.

The gold asbestos is prepared by dissolving pure gold in *aqua regia*, and evaporating the solution until nearly the whole of the free acid has been expelled. Fine threads of purified asbestos are soaked in this solution, and, after the liquid has drained off, heated in a porcelain crucible in a current of hydrogen for about fifteen minutes, when the reduction will be complete. Finally, the asbestos is washed with dilute hydrochloric acid and hot water, and dried.

In illustration of the accuracy of the method, the authors quote the following analyses among others: To a litre of pathological urine, which, according to the mean results of numerous analyses, contained 20 decimilligrammes of mercury, 30, 40·5,

and 67 decimilligrammes were added, and the amounts respectively found were 60, 55, and 77·5 decimilligrammes. C. A. M.

INORGANIC ANALYSIS.

The Analysis of Tungsten Compounds. F. Ibbotson and H. Brearley. (*Chemical News*, vol. lxxx., pp. 293, 294.)—On adding strong nitric acid, drop by drop, to a boiling solution of lead tungstate until the colour begins to turn yellow, then running in a few c.c. of the acid, and boiling a short time, a precipitate of tungstic acid is obtained corresponding in weight with the known quantity present. As, however, from 2 to 3 milligrammes of tungstic acid are found in the filtrate, the accuracy of the method depends on small compensatory errors. The colour change not being developed in presence of ammonium salts, the latter must be previously eliminated with caustic alkali.

Again, the acid lead tungstate thrown down by lead acetate from faintly (acetic) acid solutions can be dissolved in warm concentrated hydrochloric acid, and tungstic acid completely precipitated therefrom by the addition of water. No interference in this precipitation is exercised by barium, strontium, calcium, magnesium, zinc, cadmium, manganese, nickel or cobalt. In the case of *arsenic*, a few grammes of ammonium nitrate are added before precipitating the tungsten; any lead arsenate thrown down is redissolved in the hydrochloric acid, and may be disregarded, as may be also the precipitate formed with tungstates by *mercury*.

Uranium forms insoluble tungstates, which can be dissolved in a slight excess of sodium carbonate, and the clear solution should be heated to near the boiling-point and then poured into 6 or 7 c.c. of hot acetic acid, with an excess of lead acetate, the whole being well shaken. After just boiling the mixture the precipitate is washed by decantation, etc., and will then be free from uranium.

Iron comes down with the lead tungstate precipitate. To remove it the mixture is rendered faintly alkaline and then slightly acidified with acetic acid, the bulky precipitate furnished by lead acetate being washed thoroughly by decantation, ignited, and treated with hydrochloric acid as though it were pure lead tungstate. Small quantities of aluminium can be similarly dealt with.

Tin and *silicon* prevent the accurate precipitation of tungsten by this method, the former producing compounds insoluble in hydrochloric acid, whilst the silico-tungsten compounds are imperfectly thrown down by lead salts. They may, however, be precipitated by mercurous nitrate, from a neutral solution, the silica being removed by hydrofluoric acid after igniting the residue. C. S.

A New Method of Separating the Gadolinite Earths, and of Obtaining Pure Yttria. W. Muthmann and R. Böhm. (*Berichte*, 1900, xxxiii., 42-49.)—This is based on the fact that the normal chromates of the rare earths are considerably more insoluble than the corresponding sulphates. On precipitating a dilute neutral solution of the salts of the cerite or gadolinite earths with potassium chromate, the precipitates, when crystalline, have a composition corresponding to the general

formula $R_2(CrO_4)_3 + nH_2O$. The amount of water varies with the temperature and the metal, but most of these chromates crystallize with 8 molecules.

The authors have found that the most rapid method of fractionally precipitating these rare earths is to treat their readily soluble dichromates with potassium chromate. For the separation of the commercial yttria material into its six or more components they have proved that the following precautions are necessary:

1. The solutions of the rare earth salts and of potassium chromate must both be very dilute.

2. The liquid must be kept boiling throughout the precipitation.

3. The resulting precipitates must be kept in a fine state of division, and brought into intimate contact with the liquid. In carrying out these conditions they heat the liquid to be precipitated in a large retort nearly to the boiling-point, and then conduct a current of steam into it, the neck of the flask being slanted upwards to avoid loss by spurting. Besides the steam tube a second tube provided with a tap passes through the tubulure of the retort, and is connected by means of a siphon arrangement with the potassium chromate solution which is contained in a graduated flask. As each drop falls from the tube the resulting precipitate is distributed throughout the rapidly-boiling liquid. After the precipitation of each fraction the liquid is evaporated to its original volume, so that the conditions are the same throughout.

The following experiment is described to illustrate the method: The mixture of earths used was bought as "Yttrium oxydat. pur.," but in reality was found by spectrum analysis to contain oxides of most of the other rare earths. Forty grammes of this were triturated with about 90 grammes of chromium trioxide, and the mixture treated with a litre of water. The resulting solution of the dichromates was treated with potassium chromate until a turbidity resulted, and was then transferred to the retort and precipitated into six fractions as described above.

Fraction I. precipitated in two hours by 250 c.c. of 10 per cent. potassium chromate solution yielded only 1·1 gramme of oxides owing to the solution still containing polychromates. The oxides contained about 25 per cent. of erbian earths.

Fraction II(a). obtained in the same manner yielded 13·6 grammes of the oxides, of which about 20 per cent. were erbian earths. The spectrum showed that there was a considerable increase in the proportion of gadolinium as compared with the original substance.

Fraction II(b). was deposited on evaporating the mother liquid to the original volume. It yielded 6·04 grammes of oxides containing about the same amount of erbian earths as Fraction *II(a)*.

Fraction III. was precipitated with 300 c.c. of 5 per cent. potassium chromate solution and yielded 7·04 grammes of oxides, of which about 10 per cent. consisted of erbian earths.

Fraction IV., obtained in the same manner as *III.*, yielded 5·35 grammes of oxides containing only about 5 per cent. of erbian earths. On concentrating the liquid the chromate of a lighter earth was deposited. The oxide (2·065 grammes) was almost pure white, and showed only traces of erbium.

Fraction V. consisted of chromate of yttrium and yielded nearly pure yttria (4·2 grammes).

Fraction VI. gave 1·5 grammes of oxide, which was also practically pure yttria.

The residual solution, from which nothing more could be precipitated by potassium chromate, yielded on treatment with sodium hydroxide a slimy precipitate consisting of magnesia, calcium hydroxide, silica, and traces of yttria.

C. A. M.

Estimation of Calcium Carbonate in Soil. Immendorff's Process for the Determination of Total Calcium. H. Schütte. (*Zeit. angew. Chem.*, 1899, 854.)—The process already described by Stutzer and Hartleb (ANALYST, xxiv., 275) for estimating the proportion of calcium and magnesium carbonates, as distinguished from ferrous carbonate, in soil, yields perfectly accurate results when it is carried out on the pure carbonates of the alkaline earths; but when it is applied to natural soils, especially such as are poor in chalk, it leads to quite erroneous conclusions. In the first place, zeolites are present in larger or smaller quantities in most soils, and silicates of this nature react with the ammonium chloride, liberating ammonia, which neutralizes part of the standard acid placed in the receiver of the apparatus. The influence of such easily decomposed silicates is shown by the fact that if a soil giving only a slightly higher proportion of "carbonates" by distillation than by direct estimation of the carbon dioxide is repeatedly extracted with water saturated with carbon dioxide so as to dissolve and remove part of the true calcium carbonate, and is then again tested by the two processes, the difference between the yields is considerably increased. Secondly, Stutzer and Hartleb have stated that ferrous carbonate can be wholly decomposed by boiling the earth with water alone before the distillation is begun; this is quite true of fresh artificially precipitated ferrous carbonate, but the natural substance is far more stable, and, indeed, it can neither be broken up entirely in the manner indicated, nor correctly estimated by the authors' method. Moreover, the process does not give even concordant results when it is conducted according to the directions already specified; it is necessary to distil the liquid almost to dryness to ensure complete decomposition of the carbonates, or to use a larger excess of ammonium chloride; it is perhaps better to start with 25 c.c. of a 25 per cent. solution of chloride and 200 c.c. of water, distilling until only 25 c.c. of liquid are left in the retort, *i.e.*, till the residual liquid has again a concentration of 25 per cent.

Nevertheless, in the case of soils rich in alkaline earth carbonates, but free from ferrous carbonate, the method should prove of some value, for the errors are then relatively insignificant; but when the carbonates form but a small percentage of the total material it is no longer of service. For instance, a sample of marl containing 36·97 per cent. of carbon dioxide or 84·02 per cent. of calcium carbonate by gravimetric analysis gave 37·56 per cent. or 85·36 per cent. respectively on distillation; whereas a mud with 0·05 per cent. of carbon dioxide or 0·11 per cent. of calcium carbonate gravimetrically, was represented to contain 0·16 per cent. or 0·36 per cent. respectively by the Stutzer and Hartleb process.

To assume that the question whether a soil would be improved by the addition of chalk can be answered simply by noting the proportion of calcium carbonate already in it is not universally correct; earths rich in humus occasionally contain no calcium carbonate, yet they are not improved by introducing chalk. In many cases, therefore, it is desirable to estimate the total calcium, which is best done by Immendorff's process. The sample is extracted with hot hydrochloric acid, a portion of the solution is rendered just alkaline with ammonia, warmed and clarified by the cautious addition of more acid, then brought to the boil. A large excess of a strong solution of ammonium oxalate is next introduced, and after boiling some time the whole is made alkaline with ammonia, and faintly acid with acetic acid. The calcium oxalate can then be titrated, but it is preferably (if the soil contains much iron) ignited and weighed. Manganese in the precipitate is thus shown by the dark colour, and can be removed by a second precipitation, or by the use of bromine as usual. If the operation is properly conducted, iron and aluminium never contaminate the calcium oxalate. Passon's method for the estimation of calcium (*Zeit. angew. Chem.*, 1898, 776) is more troublesome to use, and does not give more accurate figures than that just outlined. F. H. L.

Separation of Chlorine and Iodine. L. Vanino and O. Hauser. (*Berichte*, 1900, xxxii., 3615-3617.)—This method is based on the behaviour of these halogens towards an alkaline solution of formaldehyde, the chloride being rapidly reduced to metallic silver whilst the iodide is not attacked.

The solution containing the mixed salts is precipitated with silver nitrate and the precipitate washed as far as possible by decantation, the washings being passed through a filter. It is then treated in the beaker with 25 c.c. of a solution of 50 grammes of potassium carbonate in 100 c.c. of water and 5 c.c. of a 42 per cent. solution of formaldehyde, and allowed to stand until carbon dioxide ceases to be evolved. Initial heating to 30° or 40° C. accelerates the reaction, which is usually complete in about thirty minutes. Any particles of chloride on the filter are also reduced to silver by repeated washing from a jet with the reagent previously heated to 40° C.

The precipitate in the beaker is washed with hot water, as far as possible by decantation, and after being treated with hot dilute nitric acid the liquid is repeatedly filtered through the same paper as used before, until the filtrate is perfectly clear. The silver iodide left upon the filter is washed, dried, ignited in a porcelain crucible and weighed.

The silver in the filtrate is precipitated with hydrochloric acid, and from the weight of silver chloride obtained the amount of the chlorine originally present can be calculated.

The results obtained in test experiments are given in tabular form, and show a close agreement with the theoretical quantities.

This method cannot be used for the separation of bromine from chlorine or from

iodine, since silver bromide is more or less reduced by the author's reagent under varying conditions, though not completely reduced like silver chloride. C. A. M.

A New Reaction for the Detection and Estimation of Nitrous Acid. **H. Erdmann.** (*Berichte*, 1900, xxxiii., 210-215.)—In the author's opinion the methods used for the detection of nitrites in water leave much to be desired. He states that in his experience well and surface water in which the nitrites correspond in quantity to a $\frac{1}{100000}$ N solution contain large numbers of bacteria. In exceptional cases the bacterial production of nitrites in natural water may reach a concentration corresponding to a $\frac{1}{10000}$ N solution. When the amount of nitrite nitrogen does not amount to 1 centigramme in a cubic metre of water, he considers that it may be ignored, but that when it amounts to from 1 centigramme to 1 gramme per cubic metre an, at least, approximate estimation is necessary in order to form a correct judgment of the character of the water.

If the nitrous acid in the water can be quantitatively converted into a coloured azo compound, it should be possible to make a colorimetric estimation. Metaphenylene-diamine, and similar substances, however, cannot be used for this purpose, since the brown colorations obtained are not sufficiently characteristic; and, on the other hand, they also give somewhat similar colorations with oxidizing agents.

After numerous experiments, the author has finally fixed upon 1-8-amido-naphthol 4-6-disulphonic acid as the most suitable substance. This is prepared from 1-3-5-naphthalene-sulphonic acid (*Berichte*, xxxii., 3186) by nitration, reduction, and heating with sodium hydroxide. It has the property of combining in acid solution with diazo compounds to form mono-azo colouring matters which are readily soluble and have a characteristic tint.

In using the reagent, 50 c.c. of the water are treated with 5 c.c. of a hydrochloric acid solution of sulphanilic acid (2 grammes of sodium sulphanilate per litre), and after about ten minutes about 0·5 gramme of 1-amido-8-naphthol-4-6-disulphonic acid in the solid state is introduced. In the presence of nitrous acid a brilliant Bordeaux red colour is produced, and reaches its maximum intensity in about an hour.

For a quantitative estimation, the colour is compared after that lapse of time with that given by standard solutions under the same conditions. The solutions used are $\frac{1}{1000000}$ N, $\frac{1}{100000}$ N and $\frac{1}{10000}$ N, and should be freshly prepared in order to avoid bacterial alteration.

As a proof of the superiority of this test over bacteriological methods, the author cites a case in which a well-water, which had given rise to an epidemic of anthrax, gave a pronounced nitrite reaction after a year, although the bacteria had long disappeared. He points out that it is seldom possible to actually isolate anaërobic pathogenic bacteria on account of their short life.

As regards other tests for nitrites, that of Griess with α-naphthylamine (*Berichte*, xii., 427) is said to be not sufficiently sharp or characteristic for water analysis, and to be unsuitable for a colorimetric estimation.

Still less satisfactory is Riegler's reagent, naphthionic acid (ANALYST, xxii., 305),

since, as is the case with many other readily diazotized bodies, its fluorescence interferes with the comparison. The author's reagent is stated not to fluoresce under the conditions given above. With oxidizing agents, such as ferric chloride, it gives a yellow coloration, which cannot possibly be confused with the red colour obtained with nitrites.

The author has made arrangements with J. F. Schwarzlose Söhne (Berlin, S.W., Markgrafenstr. 29) to forward specimens of his new reagent gratis on application.

C. A. M.

The Estimation of Sulphydric, Sulphurous, and Thiosulphuric Acids. W. Feld. (*Chem. News*, lxxxi., 40.)—The methods recommended by the author are applicable to the salts of the alkalies and of the alkaline earths.

For *sulphides* the sample is boiled with a concentrated solution of magnesium chloride in an atmosphere of carbonic acid, when (assuming barium sulphide) the following reaction takes place:

$$BaS + MgCl_2 + CO_2 + H_2O = BaCl_2 + MgCO_3 + H_2S.$$

The sulphuretted hydrogen evolved is absorbed in a solution of iodine, which is afterwards titrated.

For *sulphites* the same procedure is adopted, but substituting hydrochloric acid for magnesium chloride.

Thiosulphate is first converted into tetrathionate by titration with iodine. The solution is then subjected to the action of aluminium and dilute hydrochloric acid in an atmosphere of carbonic acid, when the following reaction takes place:

$$Na_2S_4O_6 + 20HCl + 6Al = 2NaCl + 3Al_2Cl_6 + 6H_2O + 4H_2S.$$

This method is also applicable for the determination of thiosulphate in the presence of sulphite which becomes oxidized to sulphate.

For the determination of sulphite in the presence of thiosulphate, the latter is first decomposed by the addition of mercuric chloride, thus:

$$Na_2S_2O_3 + HgCl_2 + H_2O = Na_2SO_4 + HgS + 2HCl.$$

The process recommended for sulphites is then applied.

In the case of mixtures of sulphide, sulphite, and thiosulphate, the sulphide is first determined by the magnesium chloride method, after which the thiosulphate is decomposed by mercuric chloride, and the sulphite determined by boiling with hydrochloric acid. The thiosulphate is determined in a fresh portion by titration with iodine and treatment with aluminium and dilute hydrochloric acid. H. H. B. S.

Determination of Nitrogen in Fertilizers containing Nitrates. F. P. Veitch. (*Journ. Amer. Chem. Soc.*, vol. xxi., p. 1094.)—The author modifies Field's variation of the Gunning method, in which potassium sulphide alone is used in place of potassium sulphate and sodium thiosulphate (ANALYST, xxii., 166), by adding to the nitrate in the digestion flask 35 to 40 c.c. of sulphuric acid, containing 34 grammes of salicylic acid per litre, the whole being allowed to stand in the cold until the nitrate is dissolved, whereupon 6 or 7 grammes of powdered potassium sulphide is

added, and gentle heat is applied for a quarter of an hour, followed by heating over the full flame till clear. C. S.

Valuation of Alkali Persulphates and of Hydrogen Peroxide. B. Grützner. (*Arch. Pharm.*, 1899, ccxxxvii., 705; through *Chem. Zeit. Rep.*, 1900, 21.)—About 0·3 gramme of an alkali-metal persulphate is gradually warmed to the boiling-point with 50 c.c. of decinormal arsenious acid and a few c.c. of potassium hydroxide solution, and kept hot for a short time. When cold, the liquid is faintly acidified with sulphuric acid, a large excess of sodium bicarbonate is added, and the whole is titrated with iodine and starch. One c.c. of decinormal arsenic is equal to 0·0119 gramme of sodium persulphate. Hydrogen peroxide can be similarly treated, 1 c.c. of acid being equivalent to 0·0017 gramme of H_2O_2. F. H. L.

Valuation of Clays for Brick Manufacture. W. Cronquist. (*Oesterr. Zeits. Berg. u. Hüttenw.*, 1899, xlvii., 522, *nach Teknisk Tidsskrift;* through *Chem. Zeit. Rep.*, 1899, 322.)—A mere chemical analysis of a clay is not sufficient alone to indicate its value to the brick-manufacturer; the proportion of silica in an air-dried aluminium silicate may range between 37 and 45 per cent., but a determination of this ingredient is practically useless. The amount of sand in Swedish clays varies from 47 to 72 per cent., but all are employed for making bricks; this sand, however, should be investigated for foreign matter, since the presence of mica, for instance, weakens the bricks. The air-dried sample is boiled with 1·12 hydrochloric acid till the residue is white; it is then filtered off and treated in a silver crucible with sodium carbonate solution to dissolve gelatinous silica, and the final insoluble sand is washed, ignited, and weighed. The author suggests two physical tests. A thoroughly representative sample is kneaded with water, and pressed into a wooden mould to yield a plate $20 \times 4 \times 1$ centimetres in size, on which a line exactly 150 millimetres long is drawn. After drying, the line is observed to find the distortion and contraction; a shrinkage of 8 per cent. indicates a well-binding clay. To ascertain the strength of the material, a crucible 45 millimetres high and 2·5 millimetres thick in the walls is constructed with suitable moulds; this is dried for four or six days in the air, then for two days at 100° C. It is next placed in an oven with similar crucibles made from standard clays, and burnt as usual. Finally it is broken in a specially-designed apparatus by means of lead shot. F. H. L.

A New Method of Titrating Potassium Iodide. E. Vincent. (*Journ. Pharm. Chim.*, 1899, x., 481-483.)—This is based upon the well-known fact that iodic acid acts upon the iodide in accordance with the equation:

$$6HIO_3 + 5KI = 5KIO_3 + 3I_2 + 3H_2O.$$

One gramme of the iodide under examination is dissolved in a litre of water, and 100 c.c. of the solution mixed with the same quantity of a solution of iodic acid (2 grammes per litre).

Five to ten c.c. of a standardized solution of thiosulphate, containing 2·48 grammes of thiosulphate in 100 c.c. and potassium bicarbonate in the pro-

portion of 2 grammes per litre, are measured into a beaker, and the mixed solutions of iodide and iodic acid run in from a burette drop by drop until the liquid assumes a faint yellow tint. Five-sixths of the iodine found are derived from the iodide originally present.

The potassium bicarbonate is added to the thiosulphate with the object of preventing the oxidation of the latter by the excess of iodic acid.

The following results were obtained by this method and by the more complicated method of Falières, in which the iodine is liberated by ferric chloride:

	Falières' Method. Per Cent.	Iodic Acid Method. Per Cent.
I.	96	95·87
II.	97·2	97
III.	94·5	94·4

It is essential that the thiosulphate solution should be titrated with the iodine solution, and not *vice versâ*. C. A. M.

On the Use of Iceland Spar as a Standard in Volumetric Analysis. O. **Masson.** (*Chemical News*, vol. lxxxi., pp. 73-75.)—Compact fragments weighing about 1 to 3 grammes each should be broken from a crystal of the spar, then freed from powder by rinsing with dilute acid, washed with distilled water, dried at over 100° C., and stored in a stoppered bottle.

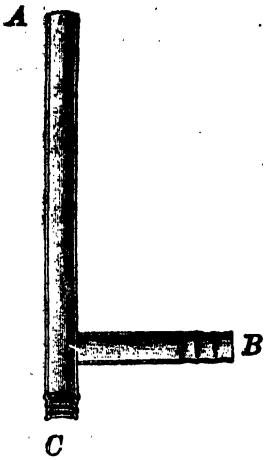
For use, about 2·5 to 3·5 grammes are weighed in an 80 to 100 c.c. beaker, the total weight alone being required, and exactly 20 c.c. of approximately normal hydrochloric acid are run in from a burette, the beaker being covered and precautions taken to prevent loss by spurting. After three to four hours the evolution of gas will have ceased, and the contents of the beaker are kept at about boiling temperature for an hour, to drive off dissolved carbon dioxide and complete the reaction. The clear solution is then decanted, and the residual lumps of spar are well washed, dried at about 110° C., cooled and weighed. The loss in weight gives the amount of spar dissolved by, and exactly equivalent to, the 20 c.c. of hydrochloric acid taken; and also furnishes direct the "factor" of the acid, since, the molecular weight of calcium carbonate being 100, 20 c.c. of strictly normal acid dissolve exactly 1 gramme.

The author discusses several possible sources of error, but regards them all as negligible. C. S.

APPARATUS.

A Mortar Cover for Use in Grinding Hygroscopic Substances. R. Scholl. (*Chem. Zeit.*, 1900, xxiv., 15.)—This consists of a rubber cap or plate with a central hole or short flexible sleeve to admit the pestle-stem, and a flange to fit air-tight round the upper edge of the mortar. The flange is made of considerable width, so that the cover is raised somewhat above the top of the mortar, thus leaving more space for the movements of the pestle. The cover is equally useful in grinding hygroscopic materials, or such as are brittle or possess an unpleasant odour.

F. H. L.



A Simple Bunsen Burner. F. Allihn. (*Chem. Zeit.*, 1899, xxiii., 996.)—In this apparatus the usual injector gas-jet attached to the base and the radial air-holes surrounding it are done away with, so as to gain simplicity and cheapness in construction. The burner consists essentially of a plain T-piece, open at all three ends. At the spot where the horizontal (gas inlet) tube joins the vertical, the wall of the latter is drilled centrally with a small hole pointing upwards towards the end where combustion takes place; and this hole being the substitute for the ordinary jet, the bore of the vertical pipe is clear throughout, so that if liquids, etc., are spilt over the burner no choking can ensue. The bottom of the upright is provided with a male thread, which screws into a cast-iron base; and the air thus enters from underneath the stand.

This latter should manifestly be cast with a milled or fluted bearing edge, or on a level table the supply of air might be insufficient.

The burner can be used equally well without the base, being supported by any convenient clamp. The apparatus is patented, and may be obtained from Warmbrunn, Quilitz and Co., Berlin. F. H. L.

REVIEWS.

LEXIKON DER KOHLENSTOFF-VERBINDUNGEN. By M. M. RICHTER. London: Williams and Norgate. Pp. 2482. (Published in thirty-nine parts, at 1s. 10d. each.)

This work is the second edition of the "Tabellen der Kohlenstoff-Verbindungen nach deren empirischen Zusammensetzung geordnet," which appeared in 1883. It contains a register of about 67,000 compounds.

Taking into account the fact that organic chemistry is a highly systematized branch of the science, the indexing of compounds by their empirical formulæ appears at first sight a retrograde step. However, that the expedient has gone far towards meeting the demands for a scheme of registering organic compounds is plain from the fact that the Executive of the German Chemical Society now publishes an index based on this system in addition to the ordinary annual index to the well-known "Berichte." A few considerations will, indeed, show that, judged on its merits, the system is the best yet proposed in many respects. Thus, even if chemists had universally agreed upon the best nomenclature for organic compounds, which is by no means the case, it must be admitted that the names denoting the constitution of chemical compounds are not in themselves well adapted to indexing purposes. They are extremely long, besides which opinions are constantly altering with regard to the constitution to be assigned to a given compound: moreover, there are the so-called tautomeric compounds to which definite constitutions cannot be assigned (*Cf.* Jacobsen and Stelzner, *Ber.*, 1898, 31, 3368). The best established property of a compound, and at the same time the one least liable to alterations, is therefore its composition which finds expression in its empirical formula.

In the system under consideration the compounds are arranged in an ascending series depending on the number of carbon atoms they contain; the elements other than carbon follow in an order which may be denoted the alphabet of the system, thus —H, O, N, Cl, Br, I, F, S, P. The remaining elements come after these in the alphabetical order of their symbols. The work includes an index of names which gives the corresponding empirical formulæ. The "Lexikon" forms a collective index to Beilstein's well-known "Handbuch der Organischen Chemie," the third edition of which has just been completed, and as such, references are given to the latter for all the compounds—about 57,000—therein dealt with. As, however, the "Lexikon" is of later date than the last edition of Beilstein, about 8,000 more compounds are referred to in the former than in the latter. In the case of very well-known compounds, besides giving the reference to Beilstein, the words "literature abundant" are added, whilst in other cases references are to be found to the principal papers dealing with the compounds in question.

It is a very common occurrence in researches in organic chemistry that in the case of certain compounds nothing beyond the composition, *i.e.*, the empirical formula, is determined. Under no other system of indexing but Richter's can these substances be registered, and consequently hitherto their record has been as good as lost. A. R. L.

SQUIRE'S COMPANION TO THE BRITISH PHARMACOPŒIA. Seventeenth Edition. London: J. and A. Churchill. Price 12s. 6d.

This work, which has attained a large circulation amongst pharmacists, is not so well known to analysts as it should be.

Although the number of examinations of drugs and their preparations made under the Food and Drugs Acts is at present small and inadequate, there is every sign that it will be increased in the near future. This being so, it behoves analysts to make a fuller study of drugs than many of them have had occasion to do in the past, and there is no work more suited to their requirements than the one under consideration.

Although this work is primarily designed for pharmacists, it contains much information of value to analysts. Thus, the normal amounts of ash are given in the case of a large number of substances; comments on official processes, with suggestions or amendments of a practical nature, are included. To cite two instances, we may take the remarks on the analysis of pepsin and the examination of malt extracts. The table in the beginning of the book dealing with the materia medica of the Pharmacopœia is of high merit, and probably the most accurate published. A prolonged study of the work serves to increase one's appreciation of the infinite care expended on the collection and presentation of the vast array of facts that have been accumulated. The writer has noticed but one misprint in the book of 850 pages.

On examining the information given below the official tests in each monograph, we find either abstracts or references to almost every recent research of importance, so that the analyst may feel confident of getting assistance in the case of nearly every drug that may come into his hands. Among the many valuable pieces of information, only two have been found that seem questionable—namely, the statements that

"nutmegs yield about 5 per cent. of ash"; and, secondly (although this is not a statement of the author's), that the difference in the petroleum spirit extracts of true and false pareira is so marked as is indicated by the difference between the figures 8 and 0·3 per cent. Analysts will find the latest edition of this work a valuable acquisition.

C. G. M.

BAYLEY'S CHEMISTS' POCKET-BOOK. Seventh Edition. London: E. and F. N. Spon. Price 5s.

The author states that this seventh edition of his well-known Pocket-Book has been rearranged and to a large extent rewritten. The book contains various numerical tables for rapid calculations, which are useful to all technical workers, whether chemists or not. The more strictly chemical part includes tables of the principal properties of the elements, various analytical factors, and tables of weights and measures, with means for translating one kind into another. We would suggest the addition of a selection of the heats of combination most frequently needed in practical calculations. Tables of specific gravity of solutions and of sundry salts are given, and are certainly acceptable, as are also records of the melting and boiling points and solubility of a number of substances, and the tables of the behaviour of salts with different indicators. Altogether, there is a great mass of matter clearly arranged and readily accessible. The fact that the book has been long before a critical public and has been received with favour implies that the data contained in it are substantially accurate. Its use may save many a search in bulkier tomes. B. B.

MILK STANDARDS.

The following letter has been received from Dr. Thorpe by Mr. P. A. E. Richards, Public Analyst for St. Martin's-in-the-Fields:

Government Laboratory,
Clement's Passage, London.
November 2, 1899.

DEAR MR. RICHARDS,

In the early days of the working of the Food and Drugs Act, when it was necessary to have regard to the character of much of the milk supplied, which, although genuine, was, as regards its fat, of no very high quality, the "standard" was fixed at 2·5 per cent. of fat.

By the operation of the Food and Drugs Act, and the weeding out of certain classes of cows, there has been a gradual improvement in the general character of the milk-supply of the country, as was shown by the investigation undertaken by Dr. Bell shortly before his resignation, and published as a Local Government Board paper.

As a result of this inquiry, I raised the standard of fat in 1894 to 2·75 per cent.

The causes above alluded to have continued to operate, as shown by the evidence taken by the Committee which more recently reported on the working of the Food and Drugs Act. In consequence of this, and in view of the fact that under Section 4 of the new Act, which comes into force on January 1, 1900, a *presumption* of non-genuineness may be raised by the analyst should the amount of fat fall below a certain figure, whereas the onus of *proof* of the genuineness will fall upon the seller, I have felt justified in raising the limit to 3 per cent., and, as you may have noticed, this number has been given in recent certificates.

This limit is, however, not to be held to anticipate or prejudge in any way the regulations as to standards which the Board of Agriculture may issue under the section of the Act referred to.

Yours very truly,
(Signed) T. E. THORPE.

THE ANALYST.

JUNE, 1900.

PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS.

THE monthly meeting of the Society was held on Wednesday evening, May 2, in the Chemical Society's Rooms, Burlington House. The President (Mr. W. W. Fisher, M.A.), occupied the chair.

The minutes of the previous meeting were read and confirmed.

Certificates of proposal for election to membership in favour of Messrs. J. R. Brooke, T. H. Lloyd, and G. H. Morris, Ph.D., were read for the second time.

Messrs. Thomas Eustace Hill, M.B., B.Sc., Thomas Macara, and William E. F. Powney were elected members of the Society.

The following papers were read: "The Composition of Milk and Milk Products," by H. Droop Richmond; "A New Colour Reaction for Citral and certain other Aromatic Compounds," by Herbert E. Burgess; and "On the Detection of Zirconia in the Presence of Thoria," by Otto Hehner.

THE DETERMINATION OF CARBON AND SULPHUR IN STEEL.

BY BERTRAM BLOUNT, F.I.C.

(Read at the Meeting, March 7, 1900.)

EXISTING methods in common use for the determination of carbon and sulphur in steel are curiously roundabout. The obvious course is to oxidize the whole mass of metal, so that all its constituents—iron, carbon, sulphur, phosphorus, manganese, and minor impurities—are brought to their highest state of oxidation consonant with the conditions, and to collect and separate those products which are volatile—viz., the oxides of carbon and sulphur. This method has been propounded with many elaborations for carbon. As a rule, the analytical description begins by requiring that the steel shall be reduced to fine powder to aid its combustion, a proceeding hopelessly impracticable in the case of mild, tough metal used for structural purposes, and sufficiently difficult with any grade of steel. An alternative plan is to remove the iron by volatilization in a stream of chlorine, which must be free from oxygen, and to burn the carbon thus disentangled from iron in oxygen. The difficulty of insuring the freedom of the chlorine from oxygen is the chief obstacle to the wider use of this method.

I have tried to work out a process for determining both carbon and sulphur by direct combustion of steel of all grades in oxygen. In this endeavour I have encountered certain difficulties which are not yet wholly overcome; but the work has reached a stage at which it may be usefully recounted. The process employed was extremely simple. The steel was heated in a porcelain tube in a current of oxygen, and the gaseous products absorbed and estimated by the usual means. As the method was intended for use in ordinary practice, no attempt was made to divide the metal finely; turnings or drillings, such as are commonly taken from a sample for analysis, were employed in all my experiments.

1. *Determination of Carbon.*—Various experiments were carried out to determine the conditions necessary to be observed in order to oxidize completely fairly thick fragments of steel, such as ordinary turnings or drillings. As it was an obvious advantage to work at a moderate temperature, the attempt was made to oxidize the steel at the temperature attainable in a porcelain tube heated by a good Fletcher combustion furnace. The temperature which can be reached thus may be taken as between 700° C. and 800° C. It was found that oxidation took place readily at first, but that the steel soon became coated with a layer of magnetic oxide so dense and adherent that the rate of oxidation rapidly fell off. This layer doubtless acts as a carrier of oxygen to the core of unoxidized metal; but the speed of translation of oxygen is so low that complete combustion cannot be effected in a reasonable time, and the process is impracticable. It is evident that the rate of oxidation would be greatly increased if the combustion could be effected in the presence of some flux which would slag off the iron oxide as fast as it was formed. A mere solvent flux such as boric acid would be useless, because the glass formed by it would protect the unoxidized metal even more effectually than the layer of magnetic oxide. The flux should be an oxidizing agent as well as a flux, and preferably one capable, not only of supplying, but of carrying, oxygen. Lead oxide fulfils these conditions; but, unfortunately, no ordinary material which can be used to make a boat will withstand the attack which it, or the lead reduced from it by the iron, may cause. Pending the construction of a boat of fused alumina or some similar material, the use of lead oxide as an oxidizing flux must be set aside.

Oxidation of the steel at a higher temperature was then tried. It was found that at a temperature of about 1,100° to 1,200° C. (well above the fusing-point of copper), such as can easily be attained in a small Fletcher lecture furnace, oxidation proceeded fairly fast, and for a quantity of 3 to 5 grammes of metal was usually complete in about one hour, reckoning from the time when the furnace became fully hot. The rate of oxidation was at first so rapid that the oxygen passing through the tube was almost completely absorbed; as soon as a crust of magnetic oxide had formed the rate naturally fell off. Occasionally there remained a slight core of metal, but analysis of this showed it to be free from carbon. The core had been decarburetted by a kind of reverse cementation.

Ultimately the method arrived at was as follows: 3 to 5 grammes of steel in the form of drillings or turnings, which may be quite thick,* are placed in a porcelain boat, and heated in a porcelain tube for at least one hour at a temperature of 1,100°

* Evidently, if thin turnings are available, they will present the advantage of oxidizing more rapidly.

to 1,200° C.—*i.e.*, to about as high a temperature as a good porcelain tube will bear. [The process of oxidation may be watched through a glazed spyhole by the use of the little device described in a paper of mine on "The Determination of Oxygen in Copper," ANALYST, xxi., 57]. The oxygen before it enters is purified by the usual train of absorption tubes, and the gases as they leave the porcelain tube pass through a glass tube kept at a dull red heat, and containing a column of copper oxide and one of lead chromate. This is necessary to insure the oxidation of small quantities of CO which might arise from a momentary defect of oxygen, and also to stop sulphur, which is the only element other than carbon in the steel yielding a gaseous oxide. Direct experiment has proved that no sulphur escapes from this tube into the absorption bulbs. The purified CO_2 is dried, absorbed, and weighed in the usual manner.

Now, although the advantages of this direct method over the usual lengthy process of dissolution in potassium cupric chloride, filtration and combustion of the residue are so manifest that their discussion would be tedious, yet there are certain difficulties and defects which arise, chiefly from the high temperature needed for oxidation. The life of the porcelain tubes is unduly short, and there is some risk of cracking in the course of an experiment. I am inclined to think that tubes—and, in fact, porcelain generally from the Royal Berlin porcelain factory—is now somewhat inferior to that which was made some years since; I have noticed many small signs of a lowering of quality. The necessarily high temperature of the air in the neighbourhood of a small but vigorous injector furnace makes the task of the absorption tubes unusually difficult, unless they are elaborately screened from radiation or kept at a distance; in the latter case the connections are inconveniently long. Lastly, it is almost a necessity for practical work that the blast of air should be obtained by power; the use of a foot-blower is too laborious, and the risk of unduly rapid heating is too great to allow of the ordinary bellows being conveniently employed. In my own case I use a Root's blower driven by a gas-engine for all such operations.

On account of these difficulties, which sometimes cause errors, I cannot recommend the method as being in its present state capable of replacing generally the older process; it is, however, useful as a completely independent method of checking a result which may appear to be in doubt. What is needed in order to make it generally applicable is some modification that will allow of the complete oxidation of the metal at a temperature not exceeding a moderate red heat—say, 800° C.

2. *Determination of Sulphur.*—This is the only other element commonly present in steel which, on the complete oxidation of the metal, yields a gaseous oxide. It seemed reasonable to suppose from general considerations that a fragment of steel heated in oxygen until fully oxidized would yield the whole of its sulphur as SO_2 (or partly as SO_3). Granting this, the estimation of the sulphur would be extremely simple, consisting merely in combustion of the steel in oxygen and reception of the products of combustion in any suitable absorbent. In my experiments I used apparatus similar to that employed for carbon. The steel, in the form of drillings, was heated in a porcelain tube to nearly 1,200° C. and a current of oxygen passed over it; the products of combustion were at once led into baryta-water. After the operation, the baryta-water, containing barium carbonate, sulphate, and sulphite in suspension, was

made acid with hydrochloric acid, saturated with bromine, and the resulting barium sulphate estimated in the usual way. But an unexpected difficulty arose. It was found easy to catch and estimate all the sulphur which was evolved as oxides, but quite impracticable to expel the last trace from the magnetic oxide left in the boat. As much as 20 per cent. of the total sulphur in the steel might thus remain; in what form it can be fixed is by no means clear. It would probably be feasible to expel this residual sulphur by fusion of the magnetic oxide with an acid oxide such as silica or boric anhydride but the proceeding would need an operation successive to and distinct from the combustion proper, and would so complicate the method as to destroy its chief ground for consideration. Moreover, it was found that any small fragment of unoxidized metal (which is harmless in the determination of carbon) means serious loss of sulphur, that element being segregated and gradually driven in towards the centre as combustion proceeds. The core, though small, may therefore contain a sensible proportion of the total content of sulphur.

These facts are well illustrated by the figures obtained in the following experiment: A mild steel containing 0·064 per cent. of sulphur was used; 10 grammes of this were oxidized; the sulphur passing into the absorption bulbs, that remaining in the crust of magnetic oxide and that remaining in the small metallic core (weighing 0·4075 grammes), were severally determined. The results are appended:

	Per Cent.	
Sulphur volatilized	0·006	
Sulphur from magnetic oxide ...	0·041	Calculated on the original steel.
Sulphur from metallic core ...	0·017	
Total sulphur...	0·064	

This particular experiment was stopped at a point when there was still a sensible weight of unoxidized core. Had it been proceeded with until oxidation was complete and the whole mass of magnetic oxide had been strongly heated for some hours, no doubt the bulk of the sulphur would have appeared in the absorption bulbs. Even then, however, an appreciable fraction would have remained with the magnetic oxide. The large quantity of sulphur left in the core becomes more evident when it is calculated on the weight of the core itself; in this instance it amounts to 0·4 per cent.

These facts go to show that, whereas the method which I have indicated may prove useful and, with some modification, convenient for the determination of carbon in steel, the corresponding method for sulphur is not likely to be found practically available.

Discussion.

Mr. Allen said that the greater number of the steels with which he had to deal contained chromium and nickel, constituents which were not taken into account in the author's investigation, but which probably would not interfere with the success of the method proposed. As a check upon the usual method of dissolving the metal in ammonio-cupric chloride and burning the carbonaceous residue, a method based on direct combustion of the steel itself would have many advantages. The chlorine method was a good one, but, as had been pointed out by the author, the chlorine must be free from oxygen, and dry, which constituted the great difficulty of the

method. He once tried to avoid this difficulty by using bromine, which could easily be obtained dry and free from oxygen. He understood that Mr. Archbutt had successfully used chromic acid for oxidizing the carbon, but he (Mr. Allen) had not been able to get sufficiently good results with this process. The method of dissolving the iron in cupric chloride was perhaps the one most suited for practical work, and if a simple direct combustion process could be satisfactorily turned to account for checking purposes, it would be of considerable value.

Mr. ARCHBUTT said that he had tried the method of direct combustion at a red heat, but had abandoned it as being too cumbrous. The chief difficulty of Mr. Blount's method, he imagined, was to find apparatus that would stand the very high temperature. He had long ago abandoned the chromic acid method because it also was too tedious, although it was sufficiently accurate if proper precautions were observed. His present method was to dissolve the steel with the double chloride of copper and potassium, burning the residue in oxygen. Results were obtained rapidly, and duplicates were very close; but it was desirable to have an alternative method based on a different principle, by which the results could be checked. The only really reliable method of determining sulphur in steel was by oxidizing the steel with aqua regia, and precipitating the sulphur with barium chloride.

Mr. ALLEN said that his difficulty with the chromic acid method had not been that it was too tedious, but that the sulphuric acid used for dissolving the chromic acid was liable to dissolve carbon dioxide.

Mr. BEVAN suggested that a correction might be obtained for this once for all by determining the quantity of carbon dioxide dissolved by the sulphuric acid in a blank experiment.

Mr. ARCHBUTT said that as, when the carbonaceous matter was placed in a flask with chromic acid, a much larger quantity of oxygen was evolved than was necessary for oxidizing the carbon, he would have expected all the carbon dioxide to be driven off. Besides, it was customary to aspirate air through the flask and tubes after the combustion was complete.

Mr. ALLEN said that he had been anxious to measure the carbon dioxide in the nitrometer instead of weighing it. It was not possible to blow air through, but the chromic acid mixture was heated until oxygen was evolved, and the carbonic acid was absorbed. The difficulty, however, caused by the solubility of the carbon dioxide in sulphuric acid prevented the satisfactory working of the process in this way.

Mr. JENKINS observed that in such a method of determining carbon, if the combustion were carried out at a lower temperature, the core of metallic iron remaining would presumably still retain some carbon: and the difficulty would be to insure, in the case of any metallic core left after combustion, that it no longer contained any carbon.

The PRESIDENT suggested that the sulphur collected in the core might perhaps be obtained by cracking the core and picking out the middle, somewhat in the same way in which the nodules of sulphide of copper were extracted from burnt pyrites.

Mr. BLOUNT said that the segregation of the sulphur in the manner described was a phenomenon of quite general occurrence; as the crust of oxide was brittle, it

was of course possible to separate the core mechanically, and to oxidize it separately, but the process would cease to be a simple one, and there would still remain the obstacle that the sulphur lurked to a comparatively enormous extent in the crust, which was a fully oxidized product. If a core were left in the carbon process, there would of course always be room for some doubt, though it did not follow necessarily that carbon was left behind. But he did not propose so to limit the process that any core remained; properly there should be no core. He would have thought that the simplicity of the use of bromine mentioned by Mr. Allen would have balanced any disadvantages which might have attended it.

Mr. ALLEN said that the experiments with bromine had been made a good many years ago, when the question of rapidity of working was not of so much consequence as at present; but as a volatilizing agent, which could be easily insured to be dry and free from air, bromine would certainly seem to be worth considering.

Mr. BLOUNT, continuing, said that 1,200° C., although appreciably above the melting-point of copper, was by no means an impracticable temperature. A good porcelain tube would certainly stand it, and the only serious difficulty was that porcelain tubes had lately been of indifferent quality and liable to crack. He was quite of Mr. Archbutt's opinion that the aqua regia method was the only one of any practical utility for determining sulphur in steel.

MAIZE OIL.

By ROWLAND WILLIAMS, F.I.C.

(*Read at the Meeting, March 7, 1900.*)

THIS oil has attracted a considerable amount of attention during the last few years, and its properties have been investigated by several well-known chemists. Among others, Archbutt has published an interesting paper on the subject (*Jour. Soc. Chem. Ind.*, vol. xviii., p. 346). Maize oil is, I understand, largely used in the manufacture of soap, especially of soft-soap, and I am informed that it is also employed in the United States for making paints and varnishes.

It was, indeed, as a possible drying oil that maize oil was first brought under my notice, and during the last two or three years I have examined a good many samples of the oil, both analytically in the laboratory and also practically on a larger scale, with a view to determine whether its drying properties are sufficiently marked to enable it to be used, either partially or entirely, as a substitute for linseed and other drying oils.

According to Lewkowitsch ("Oils, Fats, and Waxes," p. 372), "Maize oil is, notwithstanding its high iodine value, almost devoid of drying powers. No decided drying properties are imparted to it by subjecting it to the process of 'boiling,' or by addition of lead oxide. If, however, a current of air is passed through it at 150° C. [? for how long], it will, on addition of manganese borate, acquire to a small extent drying properties, and a thin film on lead dries in ten to twenty hours, but not completely." My own experiments have led me to the conclusion that, although maize

oil does possess drying properties to a certain degree, which may be increased by suitable treatment, it is not at all likely to take the place of linseed and similar oils in the manufacture of paints and varnishes.

One advantage claimed for maize oil is the comparative paleness of a film of the oil when dry, in consequence of which it is said to be more suitable than linseed oil for mixing with white and delicate pigments in general; but I have not found its slight superiority in this respect to counterbalance its undoubted disadvantage as regards slowness of drying.

I find that a thin layer of maize oil (about ½ gramme), when heated on a watch-glass to 100° C. for some considerable time, gradually increases in weight for perhaps twenty-four hours, after which it usually begins to lose slowly, until at the end of three or four days the weight is generally less than that originally taken. The maximum increase in weight does not, as a rule, exceed 1·5 per cent., whereas good linseed oil under similar conditions will generally gain at least 4 per cent.

On heating in the way described above maize oil thickens slowly, and eventually becomes solid if the oil is of good quality and the heating is continued long enough (usually two or three days); but the film thus obtained is inferior in many respects to a linseed-oil film.

In the following table I have recorded some of the figures obtained in the examination of several samples of maize oil. These were procured from most reliable commercial sources, and I have no reason to doubt their genuine character:

	Specific Gravity (15·5° C.).	KOH Absorption, per Cent.	Free Fatty Acids (as Oleic), per Cent.	Bromine Thermal Value, ° Centigrade.	Iodine Value.		Unsaponifiable Matters, per Cent.
					Hübl (15 hours).	Wijs (15 minutes).	
No. 1	·9252	18·88	3·50	23·7	123·97	124·74	1·46
No. 2	·9268	19·07	3·02	24·4	127·27	127·48	—
No. 3	·9244	18·96	3·18	23·3	122·57	123·54	1·71
No. 4	·9256	18·62	3·64	23·4	127·36	128·02	—
No. 5	·9284	19·00	2·39	23·3	120·85	122·21	—
No. 6	·9246	18·79	1·52	23·3	125·56	126·62	—
No. 7	·9262	18·74	1·50	23·2	121·99	122·68	1·66
No. 8	·9248	18·81	2·27	23·5	123·98	125·05	—

It will be observed that the iodine value was determined by Wijs' as well as by Hübl's solution, with essentially the same results.

The bromine value of three of the samples was also determined by the bromine absorption method described by McIlhiney (*Jour. Amer. Chem. Soc.*, December, 1899). The figures obtained were about 2 per cent. lower than the theoretical bromine values calculated from the iodine values.

Discussion.

The President having invited discussion,

Dr. Lewkowitsch said that he was not altogether responsible for the passage which the author had quoted. It was, in fact, itself a quotation from the original paper of Smith. It would have been interesting to ask the author whether he had examined the unsaponifiable matter. Recently, by American chemists, the unsaponi-

fiable matter of maize oil had been variously stated to consist of cholesterol and of phytosterol, but, as a matter of fact, the unsaponifiable matter of vegetable oils had been shown invariably to consist of phytosterol, and that of the animal fats and oils to consist of cholesterol. To admit, therefore, the presence of cholesterol in maize oil would be to upset a classification which was well founded on observations. According to his experience, such an admission would be erroneous.

Mr. ALLEN observed that the percentages of unsaponifiable matter were very high as compared with those of most vegetable oils.

Dr. LEWKOWITSCH said that a high percentage of unsaponifiable matter was one of the special characteristics of maize oil.

Mr. ARCHBUTT said that the question of whether maize oil was capable of being satisfactorily blown had recently been tested for him on a practical manufacturing scale, and the result was to show that it yielded an excellent blown oil without any difficulty whatever.

ON THE ASSAY OF CREOSOTE.

BY A. D. HALL, M.A.

(*Read at the Meeting, March* 7, 1900.)

BEING not infrequently called upon for advice as to the most suitable kind of creosote for the preserving of hop-poles and other kinds of timber used agriculturally, I was led to examine a number of different samples of creosote in commerce. The authorized method for the examination of creosote, introduced, I believe, by Dr. Tidy, seemed to afford considerable scope for improvement, and, after some experiment, I have adopted the following method as more rapid in execution and less liable to errors of manipulation:

1. The creosote in bulk is warmed, and well mixed till all crystals of naphthalene, etc., are dissolved.

2. A portion is dipped out into a measuring glass marked at 100 c.c., and thence transferred to the distilling flask. The glass is rinsed out with 5 c.c. of benzene, which is added to the flask. For the distillation I prefer to use a nickel flask, holding about 500 c.c., as a glass flask can rarely be used a second time.

3. To the flask is connected by a cork a straight glass tube of $\frac{1}{2}$ inch bore about 2 feet long, which serves as a condenser. A thermometer reading to 350° also passes through the cork.

4. The condensing tube passes into the receiver, a cylindrical separator of about 300 c.c. capacity, stoppered at the top and terminating in a tap at the bottom. The cylinder is graduated from the tap upwards.

5. The distillation is begun gently, then pushed till a temperature of 325° C. (600° F.) is reached, and maintained till no further distillate passes over. It may be necessary from time to time to warm the condensing tube to prevent it blocking with naphthalene.

6. When no more comes over, the volume of distillate is read off in the receiver, 5 c.c. is deducted for the benzene used, and so the percentage of "residue non-volatile at 600° F." is obtained.

7. To the receiver 100 c.c. of caustic soda solution are now added (alkali of specific gravity 1·2 is specified in the old method, but there is no objection to the use of the alkali of specific gravity 1·3, commonly used for ammonia distillations). The mixture is well shaken and put in a water-oven for an hour at least, being well shaken from time to time. Finally, it is allowed to stand till cool, the volume read, and the greater part of the caustic soda solution is drawn off by the tap. There will be more than 100 c.c., owing to the phenols absorbed.

8. The caustic soda solution is boiled for a few minutes, to expel any traces of hydrocarbon, etc., it may retain, and allowed to cool. When cold, 10 c.c. are withdrawn by a pipette, and run into the bottle of a Leffmann-Beam centrifugal milk apparatus; dilute sulphuric acid is added carefully till the alkali is neutralized and the so-called "tar acids" are liberated, then the bottle is whirled for a few minutes in the machine, allowed to cool, and the quantity of "tar acids" in the neck of the bottle read off. A preliminary calibration of the divisions on the neck of the bottle is, of course, necessary to obtain their value in c.c. From these data the percentage of "tar-acids" in the original creosote is calculated.

9. To the contents of the receiver 100 c.c. of dilute sulphuric acid (1 : 5) is now added, and the heating and shaking of 6 is repeated. The contents of the receiver are allowed to cool, and the volume of the acid extract is read off (it is greater than 100 c.c., because some of the caustic soda solution was not drawn off). Ten c.c. of this are pipetted off into a Leffmann-Beam bottle, the "tar bases" liberated by alkali, and their volume measured after whirling, just as in the last operation. After correcting for the volume of the acid solution, the proportion of "tar bases" in the original creosote is calculated. The "tar bases" sometimes solidify, making it difficult to read their volume with accuracy.

The disadvantages inherent in the old process are the necessity of complete separation of the oil from the caustic soda solution and of the "tar acids" after acidifying, and the filtration of the latter through asbestos. By taking an aliquot part of the soda solution, the losses inevitable in the attempt to collect the whole of the "tar acids" are avoided, while the use of the small bottle and the centrifugal method enables the "tar acids" to be separated and measured with accuracy and speed. The ease of the method also enables the "tar bases" to be estimated, which is desirable, because of their preservative power. In the old method the distillate, etc., has to be transferred from vessel to vessel four times before it is actually measured as "tar acids"; in the method suggested there is no such transference of a liquid that has to be finally measured.

Example.

Readings :		
Distillate	66 c.c.	67·5 c.c.
Volume of soda solution	110 ,,	110 ,,
"Tar acids" in bottle	47 ,,	47 ,,
Volume of acid solution	103 ,,	100 ,,
"Tar bases" in bottle	15 ,,	10 ,,
Calculated :		
Residue non-volatile at 600° F.	39 per cent.	37·5 per cent.
"Tar acids"	9·3 ,,	9·3 ,,
"Tar bases"	2·8 ,,	3·2 ,,

It may not be out of place here to give a brief account of some experiments instituted to ascertain in which constituent of the creosote its preservative power mainly resides, as no sound information seems to be available on the point. The "tar acids" are always referred to in creosote specifications, and they have been supposed to act by combining with albuminoids in the wood; others, again, lay stress on the injection of the heavy oils into the pores of the wood, thus mechanically preventing the entry of water, and therefore of decay; while, again, I find many users of hop-poles prefer "creosote salts," which consist practically of naphthalene.

It was decided to institute some experiments to throw light on the relative preservative powers of the various constituents of ordinary creosote, and the first trial was carried out by Mr. H. H. Cousins, M.A., of the South-Eastern Agricultural College at Wye, upon a good sample of hemp-string, which had an average breaking strain when fresh of 33 pounds.

Lengths of this string were soaked in various extracts from creosote and other pure materials, as set out below, at a water-bath temperature; the string was partly buried for two months in ordinary garden soil, dug up, and its strength redetermined.

The results set out in Table II. gave a low value either to pure phenols or the homologous phenols extracted from commercial creosote by alkali; naphthalene was little better; while the non-volatile portion of the creosote, the "tar bases" extracted from it by acid, and the bone oil, which is rich in similar bases, had preserved the string. It does not, however, follow that string will behave in the same way as wood, and other experiments were begun upon wood. In one set small blocks of wood, sawn transversely to the grain, were soaked for half an hour in 5 per cent. solutions in benzene of the constituents to be tested, the solutions poured off, and the blocks dried. One of my colleagues, Professor J. Percival, then seeded each block with a pure culture of penicillium and also with fragments of rotting wood; the blocks were kept in a moist chamber in the dark. The experiment, however, did not succeed; most of the substances entirely inhibited the growth of penicillium, etc., though the wood was repeatedly seeded, and even streaked over first with a nutrient medium.

In the other trial a series of small rods of deal were carefully selected; they were 8 inches long by $\frac{3}{8}$ inch square, and were soaked in the various materials for one and a half hours at a temperature of 90° C. They were then buried in about three-quarters of their length for ten months in ordinary garden soil, taken up, washed and slowly dried, and then broken transversely under similar conditions, with the result set out in Table III.

The results show that the creosote itself, and particularly the non-volatile constituents, had considerable preservative power, as also had the bone oil; the tar "acids" and "bases" had a real but smaller preservative power; but pure cresol had little or none.

As far as the experiments go, they seem to show that the preservative action of creosote lies in its power to fill the cells of the wood with a fixed moisture-resisting material rather than in any chemical antiseptic action; the preservative qualities of cresol and naphthalene are small, for thin pieces of wood at any rate, because the one evaporates and the other dissolves, leaving the wood open to attack. It also

seems desirable in the commercial examination of creosote to estimate the pyridine "tar bases" and rate them as of equal preservative value to the "tar acids." Further experiments on large pieces of wood are in progress, which I hope to lay before the Society at some future time.

TABLE II.

Breaking Strain of String after Two Months' Exposure (original Breaking Strain, 33 Pounds).

Treatment.	Condition.	Breaking Strain, Pounds.
Untreated	Rotten	0
With "tar acids"	Rotten	0
volatile distillate from creosote ...	Rotten	0
phenol	Rotten	0
cresol	Rotten	0
naphthalene	Partly rotten	3
"tar bases"	One in six rotten	16
bone oil	Sound	18
non-volatile residue from creosote	Sound	16
creosote in the cold	——	4
same creosote, hot	——	13

TABLE III.

Breaking Strain (transverse) of Deal Rods exposed Ten Months (original Breaking Strain, 630 Pounds).

Treatment.	Condition.	Breaking Strain, Pounds.
Untreated	Much decayed ...	... 300
"Tar acids"	A little decayed ...	... 430
"Tar bases"	Surface good ...	... 360
Naphthalene	Surface decayed, sound within	... 410
Non-volatile residue	Sound	... 570
Animal oil	Surface fair ...	... 440
Creosote	Sound	... 470
Cresol	Much decayed ...	... 230

DISCUSSION.

The PRESIDENT having invited discussion,

Dr. LEWKOWITSCH said it seemed very probable indeed that the alkaline solution would contain some proportion of the bases, which it would be important to collect and duly take into account in the analysis.

Mr. BLOUNT said that while the methods described in the paper were certainly more refined than the older ones, nevertheless the latter, when properly worked, yielded equally satisfactory results. The practical experiments of the author seemed to indicate that one of the constituents which engineers were most careful to exclude from creosote—namely, the heavy oils—possessed considerable preservative value for wood.

Dr. DYER desired to join in thanking Mr. Hall for a very interesting paper. The

latter part of the paper confirmed the views held by the late Dr. Tidy, who used to maintain that a high percentage of tar acids was by no means a necessary index of the preservative properties of creosote. Specifications used to stipulate that not more than a certain proportion of non-volatile matter should be left after distillation at 600° C.; but Dr. Tidy exactly reversed this, maintaining that at least a certain proportion of non-volatile matter ought to be present. With regard to the determination of the tar acids, he (Dr. Dyer) would like to hear whether Mr. Hall had tried, after exhausting with a large quantity of alkali, subsequently exhausting with repeated small quantities. The usual process was to exhaust with repeated small quantities of alkali, probably for the reason that, although carbolic acid (or phenol proper) dissolved in alkali of any strength, yet the higher acids—cresols, etc.—were singularly capricious, being liable, while dissolving in a certain volume of alkaline solution, to separate out if the volume of solution were increased.

Mr. ALLEN said that cresol, the immediate higher homologue of phenol, dissolved readily in a solution of sodium cresylate, so that, if there was sufficient alkali to form sodium cresylate, it would stand dilution without separation, while if there was an insufficient quantity of alkali, the cresol dissolved in it if it was strong, being thrown out of solution on dilution. If sufficient alkali were used, the various tar acids would all dissolve, but not with equal facility. It was a case of fractional neutralization, the phenol going first, as the stronger acid. But if too much alkali were used, probably some of the hydrocarbons would go into solution as well. Creosote was very largely employed for the preservation of timber-piles, piers, etc., exposed under water, which in the tropics were destroyed very rapidly owing to the action of the teredo and other worms. A method was much to be desired for the determination of acridine, which was probably the constituent most objectionable to such animal pests.

Mr. CHAPMAN suggested that the use of cork in place of wood in laboratory experiments such as those which the author had made would probably lead to more conclusive results, owing to the greater readiness with which cork would be penetrated by fungoid growths. The difficulties experienced in determining phenols by extraction with alkaline solutions were also met with in the analysis of essential oils containing phenolic bodies. In the case of clove oil, for instance, the determination of eugenol by treatment with caustic potash solution was vitiated owing to the sesquiterpene, caryophyllene, being also appreciably soluble in the alkaline liquid. With regard to the statement as to the non-occurrence of albuminoids in wood, it was, he thought, a very general belief that, but for the presence of those substances in the wood, the cellulose would undergo little or no change.

Mr. ARCHBUTT said that the author's experiments confirmed the conclusions arrived at by Mr. Boulton, and embodied in a paper read by him before the Institution of Civil Engineers in 1884. Mr. Boulton brought forward a good deal of evidence to show that the value of creosote oil for preserving timber lay in the higher and less volatile fractions, and especially in the tar bases, much of this evidence being based on the experiments of the late Dr. Tidy. That paper led to the abandonment of the late Dr. Letheby's specification, in which the phenol was regarded as the most important constituent, and the substitution for it of Dr. Tidy's specification, in which

a certain proportion of less volatile constituents was required, the exact percentage of phenol being of less importance. Personally he had found no difficulty in using a glass retort over and over again, provided that the distillation was not carried to coking. The advantage of using very strong soda was that, when the soda was mixed with dilute sulphuric acid, a supersaturated solution of sodium sulphate was produced, in which the phenols became insoluble, so that the whole of the phenols were thrown out on adding excess of acid. For removing neutral oils and naphthalene dissolved in the soda, filtration through asbestos had been recommended by Dr. Tidy, but he (Mr. Archbutt) had found it simpler to shake the caustic soda solution with ether. In estimating the tar bases he had adopted the method of distilling a separate portion down to coking and shaking with dilute sulphuric acid. The acid liquid is then evaporated to a small volume, and the bases are thrown up by adding pieces of solid stick soda or potash.

Mr. HALL said that he had a good many times tried the effect of boiling the caustic soda solution, comparing the quantities of tar acids obtained with boiled and unboiled solution ; but he had never found any notable difference, though it was possible that differences might occur in some samples, and therefore he had suggested the boiling of the solution as a precaution. He had occasionally tried, after removing everything possible by means of alkali, what could be obtained by means of acid. The effect of second and third extractions with alkali had been tried, but as far as his experience went a single treatment with alkali extracted all the phenolic substances. With regard to the question of the presence of albuminoids in wood, it was very hard to say that anything quite of the nature of an albuminoid existed in wood, but the analogy had been pressed strongly that the preservative power of creosote was due to the sort of antiseptic action which carbolic acid, for instance, had upon a substance like true albumen, namely, coagulation. There was nothing in wood exactly equivalent to true albumen, coagulable by phenol. It was the starch always that was affected. Decay was always quickest in fresh sap wood, where the cells contained a good deal of starch. He had been trying to ascertain how far the fungi penetrated the treated wood, but could not get the fungus to start into the wood except in one case, when it had been saturated with cresol, though where the other materials mentioned were present the growth was arrested at the surface.

ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

FOODS AND DRUGS ANALYSIS.

The Influence of Certain Alcohols, Acids, and Sugars on the Digestion of Proteids by Pepsin and Trypsin. E. Laborde. (*Journ. farm. Chim.*, 1899, x., 484-488.)—In his comparative experiments on this subject the author digested a definite quantity of coagulated egg albumen with acid pepsin or alkaline trypsin in the presence of 50 c.c. of a 20 per cent. and 5 per cent. solution of the given alcohol, and also as a check with the enzymes in 50 c.c. of water. The flasks were kept at a temperature of 40° C. for four hours in the pepsin digestions, and for three hours in the trypsin digestions, and at the end of those periods the amount of albumoses and peptones in the filtered liquids was determined by precipitating the former with ammonium sulphate and estimating the organic nitrogen in the solution before and after removal of the albumoses.

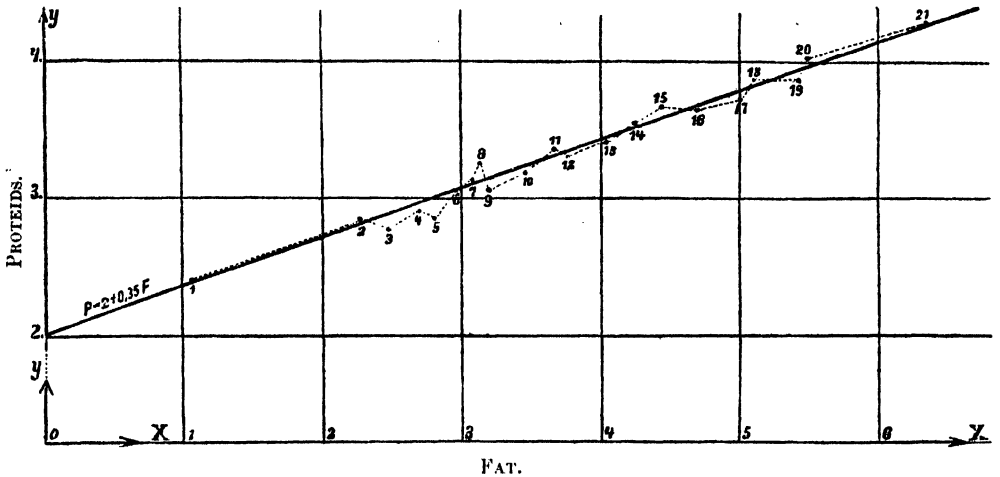
In the experiments with pepsin it was found that isobutyl alcohol, glycerol, and malic acid favoured the digestion when present in small quantity, whilst methyl alcohol appeared to have a very slight accelerating influence. On the other hand, the digestion was markedly retarded by the presence of ethyl and propyl alcohols, by lactic and tartaric acids, and by mannitol and glucose.

In the case of trypsin the activity of the enzyme was accelerated by methyl and isobutyl alcohols, by glycerol, and by glucose, and retarded by ethyl and propyl alcohols, by lactic, malic, and tartaric acids, and by mannitol. C. A. M.

The Ratio between Proteids and Fat in Milk. H. Timpe. (*Chem. Zeit.*, 1899, xxiii., 1040.)—In Table I. (p. 158) are given the analytical results obtained on examining milk derived from cows of different breeds and different localities, the samples being placed in order according to the amount of fat they contain. These figures bring to light various relationships between the several constituents in the normal secretion which are apt to be overlooked when the milk of only one description of cattle is tested, since the composition of the liquid produced by each breed is too uniform to show these ratios distinctly. Calculated per 100 parts of total solids, it will be seen that the sugar varies inversely with the fat, so that within certain limits the proportion of sugar plus fat is constant. The ash (calculated on the solids) also decreases as the fat rises, so that, with five exceptions, the value sugar divided by ash is always between 6·04 and 6·84. On the other hand, the amount of proteids (calculated on the solids) is remarkably uniform: the largest difference among the fats is 27·43 per cent.; among the sugars, 22·92 per cent.; but among the proteids it is only 2·88 per cent., and even the ash varies within wider limits. Therefore per 100 parts of total solids the amount of fat, sugar, and ash together is essentially constant. This fall in sugar and ash as the fat rises, when calculated on the solids, finds a simple explanation if the two former ingredients remain practically the same, however the fat varies, when the calculation is made upon the original milk. The table shows signs

of this: the fat in No. 21 is 520 per cent. of that in No. 1; the greatest increase in sugar is only 13 per cent., and that in ash 26 per cent. of their lowest proportions respectively. In contradistinction to the fat, therefore, it may be said that the amount of sugar and ash in milk is roughly constant.

Matters are different in the case of the proteids. If these were constant in amount in the milk itself, they should fall, like the sugar and ash, as the fat rises when calculated on the solids. It appears, however, that they are remarkably constant in the total solids; therefore they must increase (calculated on the milk), though on a far smaller ratio, with increases in the fat. Although there is no ratio between the percentage of proteid and of fat in the milks, there is a distinct ratio



between their several variations; for it will be observed that when the latter rises 5·36 per cent., the former rise 1·89 per cent., which is roughly 3 : 1; and this ratio obtains not only between the extreme samples, but also practically throughout them all. It is evident, therefore, that the ratio may be expressed by the formula $P = a + bF$, where P is proteid, F fat, and a and b two constants. From Table I. a is shown to equal 2, b to equal 0·35; and the formula thus becomes:

$$P = 2 + 0{\cdot}35F.$$

The figures in column 8 record the amount of proteid calculated from the fat by this formula, and column 9 gives the differences between the theoretical and analytical results. The coincidence is brought out more clearly in the accompanying diagram, where the thick line marks the theoretical value of P. The maximum difference is ±0·12 per cent.; only in three cases does it exceed ±0·08 per cent.

It may also be imagined that the proteids are divisible into two classes, α and β. The β-proteid perhaps is formed from the same original substance which yields the fat, and the ratio β-proteid : fat equals 0·35 : 1. The α-proteid is produced independently of the fat, and is present constantly in the proportion of 2 per cent. Thus, it may be said generally that ash, sugar, and α-proteid are approximately constant in normal milk; β-proteid and fat vary in the ratio mentioned.

These observations apply only to the milk of healthy cows fed with normal food;

illness and exceptional feeding lead to different results. In Table II. (p. 159) are collected various more or less abnormal milks. Nos. 1 to 3 are from stall-fed cattle; Nos. 4 to 8 from experimentally-fed cows; No. 9 is not described; Nos. 10 to 12. represent the milk of a cow suffering from foot-and-mouth disease; No. 13 onwards record the history of a Simmenthal cow ill, and recovering, from a severe attack of gastric catarrh ("Magenkatarrh"). [The ash figures are omitted; they appear unaffected except in Nos. 10 to 12, where they stand at 1·01, 0·88, and 0·90 per cent. respectively.] With the exception of No. 9, special feeding has caused the amount of proteids to be less than the theoretical—that is to say, has produced an excess of fat, which is the more noticeable seeing that the fat and one portion of the proteids have a common origin. This increase of fat must therefore be regarded as abnormal, if not pathological—a view which is strengthened by the phenomena of foot-and-mouth disease and gastric catarrh in its acute stages (Nos. 10 to 16). The sudden change from a deficiency to an excess of proteids on the fourth day of the latter ailment may be explained by the complete alteration in character of the milk during disease, which causes the ordinary ratios to be no longer valid; by November 6, when the catarrh had disappeared and only the animal's body-weight was still reduced, the secretion may be said to have become normal again. It should be noticed that, with five exceptions, even when the cows were ill, the amount of sugar remained within its proper limits of 4·4 to 5 per cent. This matter is of great importance in the valuation of milk samples.

To show that the ratio of fat to proteids is a constant, and is affected only by disease or by sudden changes in the conditions of life, but not by the influence of the food itself, some experiments made by Landbeck may be quoted. Three cows were fed specially during four periods of twenty days with a diet rich in proteid and fat. The proportions of the latter in the food were higher during the second period than during the first, higher during the third than during the second; during the fourth they were the same as at first. Each period was divided into a preliminary and a second stage of ten days each (the food being the same in both stages of the same period). One of the cows remained practically constant in weight throughout the whole experiment, having so good a constitution as not to be deranged by the sudden alterations in diet. The analyses of its milk were as follows:

		Proteids.		
	Fat.	Found.	Calculated.	Difference.
Period I., first stage	4·20	3·13	3·47	−0·34
second stage	4·10	3·40	3·44	−0·04
Period II., first stage	4·29	3·33	3·50	−0·17
second stage	4·15	3·44	3·45	−0·01
Period III., first stage	4·52	3·49	3·58	−0·09
second stage	4·29	3·51	3·50	−0·01
Period IV., first stage	4·35	3·43	3·52	−0·09
second stage	4·32	3·45	3·51	−0·06

It will be evident that it was the sudden changes in food, and not the food itself, which affected the ratios, for otherwise the "differences" which rose at each alteration would have remained of the same dimensions during the second stage of each period. The other two cows gained in body weight during the investigation, and their milk was constantly abnormal,

Inasmuch as commercial milk is always a mixed product derived from a large number of cows, the disturbing influence on the ratio of proteids to fat which is produced by the stall-feeding of a few individuals is practically eliminated; and it may be said that the difference (D) between the proteids obtained by analysis and by calculation (calculated amount deducted from that recovered by analysis) in such mixed milk will scarcely ever exceed ±0·06 per cent. The factor thus becomes of great value in judging the quality of ordinary milk samples; and the effect of the various methods of sophistication may consequently be studied.

Removal of Cream.—If cream has been removed from a milk, D becomes positive, while sugar and ash remain normal. When, for example, milk No. 11, Table I., was skimmed till it contained only 3·02 per cent. of fat, it gave on analysis 11·74 per cent. of total solids, 4·63 per cent. of sugar, 0·74 per cent. of ash, and 3·35 per cent. of proteids. The theoretical proteids were 3·01 per cent., therefore D equalled +0·34. From the amount of proteids found, the original proportion of fat (or the degree of fat removal) could be calculated by the formula $F = \frac{P - 2}{0{\cdot}35}$, and gave 3·85 per cent.—an error of 0·13 per cent.

Addition of Water.—When the proportion of fat falls through natural causes, the β-proteids are reduced correspondingly, while the α-proteids remain unaltered; but if water is added, all the constants are lowered, and their ratios one to another are the same as in whole milk. Addition of water thus causes D to be negative. A negative D, however, by itself does not prove addition of water; it is necessary to consider the proportion of sugar and ash. By adding fat to a skimmed milk, a negative D is produced, the fat and sugar remaining normal; dilution with water would diminish the percentage of the latter bodies as well. On mixing 100 c.c. of milk No. 11 with 20 c.c. of water, the following figures were obtained: 10·37 per cent. of total solids, 3·13 per cent. of fat, 3·84 per cent. of sugar, 0·63 per cent. of ash, 2·77 per cent. of proteids by analysis, 3·10 per cent. by calculation, D being accordingly −0·33. By using the formula $\frac{-D \times 100}{P - 0{\cdot}35\ F}$, the amount of added water could be deduced, and gave 19·7 c.c.

Removal of Cream and Addition of Water.—Detection of this method of sophistication is more difficult, but in the majority of cases it can be effected with certainty. As the former process yields a positive D and the latter a negative one, if the exact amount of water is added which compensates for the removal of fat, D remains at its proper value; but as the quantity of fat removed cannot be ascertained except by analysis, it is impossible for a milk-dealer to know how much water to add, and probably D will be either too high or too low. If, however, the two processes were carried out successfully, so that D gave no sign, the deficiency in ash and sugar would be apparent. Only if the degree of dilution were so small that the percentage of sugar remained above the minimum would detection be impossible; but if the analytical results are recalculated to a basis of an average sugar-content, the figures for fat and proteids so obtained will show the removal of cream. For example, Milk No. 11 was skimmed and mixed with one-sixth its volume of water. On analysis it showed 10·36 per cent. of total solids, 2·93 per cent. of fat, 3·93 per cent. of sugar, 0·64 per cent. of ash, 2·86 per cent. of proteids (found), 3·03 per cent.

by calculation. D was equal to −0·17, for too much water had been used, the presence of which was also indicated by the lowness of the sugar. Recalculating to a basis of 4·7 per cent. of sugar, the fat was 3·50 per cent., and the proteids 3·42 per cent. Calculating the proteids from the 3·50 per cent. of fat gave 3·23 per cent., D being +0·19. This proved the removal of cream. By the formulæ given above, the added water was represented to be 19·6 (instead of 16·7) c.c., and the original proportion of fat to be 4·05 (instead of 3·72) per cent. Therefore the falsification can not only be detected, but even estimated with some degree of accuracy—at any rate, more safely and conveniently than by "an appeal to the cow."

In practice the following cases have to be considered: (1) Sugar and ash normal. If D is less than ±0·06, manipulation cannot be detected. If D exceeds +0·06, cream has been removed, the original amount of fat being $\frac{P-2}{0{\cdot}35}$. If D exceeds −0·06, the milk has been loaded with cream. (2) Sugar and ash at or below the minimum. The amounts of fat and proteids are recalculated to the normal basis of 4·7 per cent. of sugar, in order to yield their respective proportions before addition of water. If then the recalculated D is approximately correct, only water has been added; if D is positive, cream has been removed as well, and the magnitude of the sophistication can be ascertained as above.

[Judged solely on these rules, the average normal morning milk quoted by Richmond (ANALYST, 1899, xxiv., 199) would appear as though it had been partly skimmed, for it gives D = +0·226; the evening milk gives D = +0·025, and accordingly would be regarded as genuine. Even if the morning and evening figures are combined, D = +0·125, and the mixed milk would be regarded as skimmed.—ABS.]

TABLE I.—NORMAL MILKS.

Number.	Description of Cow.	Total Solids.	Fat.	Sugar.	Ash.	Proteids. Found.	Proteids. Calculated.	Difference.	Sugar÷Ash.	Per 100 parts of Total Solids. Fat.	Sugar.	Ash.	Proteids.
1	Friesland	8·45	1·03	4·41	0·65	2·37	2·36	+0·01	6·84	12·19	52·19	7·63	27·97
2	,, ...	10·60	2·28	4·75	0·76	2·81	2·80	+0·01	6·25	21·51	44·81	7·17	26·51
3	,, ...	11·00	2·51	4·96	0·77	2·76	2·88	−0·12	6·44	22·82	45·09	7·00	25·09
4	Unknown	11·42	2·75	4·99	0·76	2·92	2·96	−0·01	6·54	24·06	45·66	6·67	25·60
5	Friesland	11·04	2·84	4·61	0·71	2·88	2·99	−0·11	6·49	25·72	41·75	6·43	26·08
6	Holland	11·06	2·98	4·43	0·62	3·03	3·04	−0·01	7·11	26·92	40·07	5·63	27·40
7	Friesland	11·40	3·03	4·53	0·75	3·09	3·06	+0·03	6·04	26·58	39·73	6·58	27·10
8	"Deutsche Landkuh"	11·70	3·07	4·78	0·64	3·20	3·08	+0·12	7·42	26·28	40·87	5·51	27·36
9	"Angler"	11·52	3·12	4·69	0·69	3·02	3·09	−0·07	6·79	27·08	40·71	5·99	26·22
10	Mixed Milk ...	11·98	3·44	4·71	0·66	3·17	3·20	−0·03	7·13	28·72	39·32	5·50	26·46
11	Harz	12·44	3·72	4·63	0·74	3·35	3·30	+0·05	6·26	29·91	37·21	5·95	26·93
12	,, ...	12·35	3·77	4·49	0·78	3·31	3·32	−0·01	5·76	30·53	36·36	6·31	26·80
13	Westerwald ...	13·19	4·02	4·98	0·77	3·40	3·41	−0·01	6·46	30·48	37·76	5·84	25·78
14	Harz	13·00	4·25	4·52	0·73	3·50	3·49	+0·01	6·23	32·71	31·77	5·58	26·95
15	Freiburg	13·46	4·44	4·68	0·70	3·63	3·55	+0·08	6·68	33·00	34·78	5·21	26·98
16	Westerwald ...	13·90	4·67	4·88	0·78	3·62	3·63	−0·01	6·25	33·60	35·11	5·61	26·04
17	Freiburg	14·06	5·00	4·59	0·76	3·71	3·75	−0·04	6·00	35·56	32·65	5·43	26·39
18	Guernsey	14·71	5·13	5·00	0·69	3·83	3·80	+0·03	7·23	34·88	34·00	4·70	26·01
19	,, ...	14·59	5·44	4·60	0·72	3·83	3·90	−0·07	6·40	37·29	31·53	4·94	26·25
20	Jersey	14·90	5·52	4·68	0·69	4·01	3·93	+0·08	6·82	37·05	31·41	4·60	26·91
21	,, ...	16·13	6·39	4·72	0·76	4·26	4·24	+0·02	6·21	39·62	29·27	4·71	26·40

TABLE II.—ABNORMAL MILKS.

No.	Description.	Total Solids.	Fat.	Sugar.	Proteids.		Difference.
					Found.	Calculated.	
1	Stall-fed...	11·63	3·62	4·28	3·04	3·27	−0·23
2	,, ...	12·06	3·71	4·69	2·98	3·30	−0·32
3	,, ...	12·15	3·88	4·52	3·10	3·36	−0·26
4	Experimental feeding	11·66	3·30	4·66	3·03	3·16	−0·13
5	,, ,, ...	11·91	3·73	4·58	2·89	3·31	−0·42
6	,, ,, ...	12·41	4·10	4·36	3·19	3·44	−0·25
7	,, ,, ...	13·46	4·50	5·11	3·04	3·58	−0·54
8	,, ,, ...	15·69	6·30	4·80	3·86	4·21	−0·35
9	?	11·75	2·96	4·15	3·90	3·04	+0·86
10	Foot and Mouth Disease ...	17·77	8·76	5·35	2·65	5·07	−2·42
11	,, ,, ,, ...	19·81	9·18	7·06	2·70	5·21	−2·51
12	,, ,, ,, ...	20·61	10·00	6·90	2·81	5·50	−2·69
13	Oct. 31. Cow suffering from gastric catarrh ...	15·32	7·29	3·80	3·56	4·55	−0·99
14	,, ,, ,, ...	13·71	5·20	4·16	3·29	3·82	−0·53
15	Nov. 1. ,, ,, ...	12·60	4·05	4·61	2·23	3·42	−0·19
16	,, ,, ,, ...	13·08	4·45	4·52	3·36	3·56	−0·20
17	Nov. 2. ,, ,, ...	12·11	3·41	4·68	3·35	3·19	+0·15
18	,, ,, ,, ...	12·57	3·87	4·43	3·55	3·36	+0·19
19	Nov. 3. ,, ,, ...	12·46	3·15	4·75	3·82	3·10	+0·72
20	,, ,, ,, ...	12·50	3·64	4·54	3·58	3·27	+0·31
21	Nov. 4.	12·82	3·65	4·79	3·65	3·28	+0·37
22	,,	12·85	3·92	4·58	3·58	3·37	+0·21
23	Nov. 5.	12·34	3·29	4·64	3·62	3·15	+0·47
24	,,	12·33	3·72	4·37	3·51	3·30	+0·21
25	Nov. 6.	12·22	3·52	4·54	3·46	3·23	+0·23
26	,,	12·22	3·88	4·30	3·33	3·36	−0·03
27	Nov. 7.	12·78	4·15	4·66	3·20	3·45	−0·25
28	,,	12·78	3·95	4·86	3·30	3·38	−0·08
29	Nov. 8.	12·61	3·69	4·96	3·24	3·29	−0·05
30		12·29	3·47	4·85	3·30	3·22	+0·08
31	Nov. 9.	12·42	3·69	4·73	3·34	3·29	+0·05
32	,,	12·52	3·73	4·80	3·33	3·31	+0·02
33	Nov. 10.	12·53	3·80	4·75	3·19	3·33	−0·13
34	,,	12·50	4·18	4·45	3·19	3·46	−0·27
35	Nov. 11.	12·44	3·72	4·63	3·35	3·30	+0·05
36	,,	12·95	4·09	4·78	3·46	3·43	+0·03
37	Nov. 14.	12·72	3·60	4·95	3·49	3·26	+0·23
38		12·98	3·76	5·06	3·47	3·32	+0·15
39	Nov. 18.	12·04	3·20	4·64	3·48	3·12	+0·36
40	,,	12·15	3·28	4·79	3·42	3·15	+0·27

F. H. L.

ORGANIC ANALYSIS.

Iodometric Determination of Small Quantities of Carbon Monoxide. **L. P. Kinnicut and G. R. Sanford.** (*Journ. Amer. Chem. Soc.*, vol. xxii., pp. 14-18.)—The apparatus used is a modification of that employed by Nicloux. Twenty-five grammes of purified iodine pentoxide are placed in a small U-tube, supported in an oil-bath at 150° C., and connected to a Wolff blood-absorption tube containing 0·5 gramme of potassium iodide in 5 c.c. of water. The air to be examined is freed from unsaturated hydrocarbons, hydrogen sulphide, sulphur dioxide, and similar reducing gases by passing it through U-tubes containing sulphuric acid and solid caustic potash respectively, and is then passed through the iodine pentoxide tube, the liberated iodine being retained in the Wolff tube and estimated by $\frac{1}{1000}$ N sodium thiosulphate. The rate of flow of the air-current is regulated by dry mercury to about ½ litre per hour. The method will detect as little as 0·0025 per cent. by volume of carbonmonoxide in air. C. S.

The Separation of Formic, Acetic, Propionic, and Butyric Acids by Haberland's Method. **J. Schütz.** (*Zeit. anal. Chem.*, 1900, xxxix., 17, 18.)—The method of separation proposed by Haberland (*Zeit. anal. Chem.*, 1899, 217) consisted in liberating the acids by means of phosphoric acid, distilling them in a current of steam, and evaporating the distillate to dryness with an excess of lead oxide. The basic lead propionate, being insoluble in water, was removed; the filtrate, after removal of the lead, was evaporated to dryness with zinc oxide and the zinc acetate, and butyrate separated from the zinc formate in virtue of their different solubilities in alcohol.

In examining this method, the author has made experiments with the three acids separately by evaporating on the water-bath titrated amounts with an excess of zinc oxide and a small quantity of zinc sulphate, liberating and distilling the acids from the residue, and again titrating.

The loss increased with the molecular weight of the acids, and amounted to 10·5 per cent. with the formic acid, 73·9 per cent. with the acetic acid, and 82·3 per cent. with the butyric acid.

Similar, though somewhat more favourable, results were obtained in the case of the lead salts of these acids, and the author therefore concludes that these bases are not suitable for the estimation of these acids by the method described by Haberland.

C. A. M.

The Composition of Geranium Oils of Different Origin. **Jeancard and Satie.** (*Bull. Soc. Chim.*, 1900, xxiii., 37-39.)—The samples examined by the authors were selected from oils distilled in 1898 and 1899, and were known to be pure.

A direct determination of the esters by the ordinary method gave the following results, calculated as $C_{12}H_{20}O_2$: Cannes oil, 19·11; Spanish oil, 23·03; Corsican oil, 21·07; African oil, 23·03; Bourbon oil, 25·95; and Indian oil, 15·05 per cent.—figures in substantial agreement with those of Charabot, Dupont, and Pillet (*cf.* ANALYST, xxii., 193).

Taking into account the fact that geranium oil invariably contains free acids, the authors determined the degree of acidity in the following manner: Three grammes of the oil were mixed with 10 c.c. of 96 per cent. alcohol and 10 c.c. of semi-normal potassium hydroxide solution. After two minutes the oil was precipitated by the addition of water, and the excess of alkali titrated with standard sulphuric acid.

It is essential not to prolong the contact of the alkali with the oil beyond the two minutes, since otherwise a partial saponification takes place. Under the conditions given above the author considers that there is hardly any saponification, and that the figure obtained in milligrammes of potassium hydroxide per gramme o oil is equal, or but slightly higher than that corresponding to the free acids actually present.

In the case of a sample of African geranium oil the results thus obtained were: After two minutes, 42·93; after five minutes, 43·06; ten minutes, 43·86; twenty minutes, 45·73; forty minutes, 48·53; one hour, 49·35; two hours, 49·93; and by hot saponification, 65·80 milligrammes.

By this method of cold saponification the authors have found that geranium oil becomes oxidised on contact with the air. Thus, a sample of Bourbon oil with a cold saponification value of 56·00 gave a value of 66·73 after being left for two months in a partially filled flask. On the other hand, a Cannes oil became oxidised much less readily, the figures only varying in a year from 26·6 to 29·66.

In the subjoined table of the characteristics of the different varieties of geranium oil the esters were calculated as $C_{12}H_{20}O_2$ (allowance being made for the free acids) and the alcohols as $C_{10}H_{18}O$:

Oil of Geranium.	Density at 15° C.	Rotatory Power at 15° C. in 100 mm. tube.	Saponification Value.		Esters, per Cent.	Alcohols, per Cent.
			Hot.	Cold.		
Cannes ...	0·8972	−9·40	54·60	26·60	9·80	61·31
Spanish ...	0·9073	−7·30	65·80	43·40	7·84	66·23
Corsican ...	0·9012	−8·00	60·20	40·13	7·00	68·55
African ...	0·9006	−8·06	65·80	42·93	8·08	63·19
Bourbon ...	0·8905	−8·20	74·00	56·00	6·65	71·28
Indian ...	0·8960	−0·48	43·00	9·60	11·30	84·62

The solubility of the different oils in dilute alcohol was practically the same, 1 volume of oil dissolving in 0·9 to 1 volume of 80 per cent. alcohol, or in 2 to 2·3 volumes of 70 per cent. alcohol at 15° C.

The authors consider that it may be possible to base a method of distinguishing between oils from different sources on the fact that those containing much free acid undergo oxidation more rapidly than oils containing a smaller amount, such as palmarosa oil (Cannes oil). C. A. M.

Detection of Ionone in Violet-scented Preparations; and Separation of the Two Varieties thereof. R. Schmidt. (*Zeits. angew. Chem.*, 1900, 189.)—If the original substance is an alcoholic solution, it is poured into twenty times its volume of water, and extracted twice with ten volumes of ether; the ether is washed two or three times with water, filtered through a double paper, and distilled off. If the

substance is a solid, it may be thoroughly distilled in a current of superheated steam as long as any volatile matter passes over, extracting the distillate with ether. Pomades may be treated with warm 80 per cent. alcohol two or three times, separating the fat by solidification, then distilling the alcoholic extract (after removal of the solvent) as before. The crude material thus obtained is ready for quantitative analysis, or it can be purified for qualitative purposes only by a distillation at a pressure of 12 millimetres, collecting the liquid which comes over between 125° and 135° C.

Melting-point Determination.—0·5 gramme of the oil is mixed with a cold solution of 0·5 gramme of parabromophenylhydrazine in 2·5 grammes of glacial acetic acid. According to the amount of ionone present, the mass either solidifies directly, after an hour or two, or after the addition of a drop of water; the crystals are dried on a porous tile. The melting-point of the parabromophenylhydrazone of crude β-ionone lies between 110° and 115°; the presence of α-ionone may raise the observed temperature to 140°. By repeated recrystallization from hot alcohol or light petroleum the α-ionone derivative may be isolated, and will give the proper melting-point of 142° to 143°. Contamination of the ionones with other ketones or aldehydes will either greatly lower the melting-point, or prevent the formation of crystals.

Separation of the Ketones from other Substances (Von Baeyer's Process).—Fifty grammes of the oil are agitated mechanically for ten hours with 83 grammes of *p*-hydrazobenzene sodium sulphonate dissolved in 500 c.c. of water, and 2 grammes of strong sulphuric acid diluted with 50 c.c. of water. The mass is made alkaline with 3 grammes of anhydrous sodium carbonate, and mixed with enough ammonium sulphate (200 or 250 grammes) to cause the liquid to separate into three layers when treated with ether. The whole is extracted twenty times with ether, and the two aqueous layers are distilled with 150 grammes of phthalic anhydride in steam, taking up the volatile oil in ether. The solvent is boiled off under reduced pressure, and the residue is fractioned at 12 millimetres pressure, retaining the distillate between 125° and 135°, which contains the ionone. This process is not quantitative.

Separation and Identification of α- and β-Ionone.—This method is the one described in *Ger. Pat.*, 106, 512 [*cf. Eng. Pat.*, 1944, 1899; *J. Soc. Chem. Ind.*, 1900, 69—Abs.]. If only a qualitative identification of the two ketones be desired, the operation may be conducted on the crude oil recovered as above; but if a quantitative separation be necessary, the product of Von Baeyer's process must be used. Seventy-five grammes of commercial bisulphite solution are warmed on the water-bath, neutralized with 10 per cent. soda solution (about 25 grammes), and acidified again with 10 or 12 grammes of bisulphite. This mixture is boiled under an inverted condenser with 15 grammes of alcohol and 25 grammes of ionone for ten to twenty-five hours, according to the purity of the ketone, until a small sample of the liquid gives only a faint turbidity with water. It is then diluted with 2 volumes of water, the undissolved ionone extracted by three agitations with ether, and the aqueous portion distilled in a current of steam as long as oily drops pass over, and until the contents of the retort begin to froth. The β-ionone in the distillate is collected with ether; to the retort are added 30 grammes of sodium hydroxide, and the α-ionone recovered by distillation as before. To identify the α-ionone, its parabromophenylhydrazone is

prepared, which melts before purification at 138° to 140°, after recrystallization from hot alcohol at 143° or, if heated fast, sometimes at 145° C. It crystallizes from petroleum in hexagonal plates, from alcohol in rhomboids. The β-ionone is identified by means of its semicarbazone, which, from alcohol, gives firm white crystals, becoming yellowish in air, and melting at 148°. The compound is readily broken up by warming with dilute hydrochloric acid, and the β-ionone may be converted into its parabromophenylhydrazone, which crystallizes in large rhombs from alcohol, and melts at 118° C.

If the mixed ketones consist mainly of β-ionone, as will be shown by the first melting-point test coming out between 110° and 116°, a qualitative separation may be effected more rapidly as follows: 10 grammes of the original oil fraction are mixed with a solution of 7 grammes of semicarbazide hydrochloride and 10 grammes of sodium acetate in 40 c.c. of water, and sufficient alcohol to insure dissolution; and the whole is allowed to rest twelve or fifteen hours. It is then precipitated with water, the semicarbazone collected, washed with water, and dissolved in about 50 grammes of spirit. After a time the bulk of the β-ionone derivative crystallizes out, and much of the remainder can be recovered by stirring with a rod, starting the crystallization again with a crystal from the first yield. This β-ionone-semicarbazone is filtered off, washed with weak alcohol, and recrystallized; it melts at 148° C. The alcoholic mother liquors containing the α-ionone are warmed for two minutes on the water-bath with a little dilute sulphuric acid, poured into water, and the oil collected by means of ether. From this the parabromophenylhydrazone of α-ionone may be prepared, and recognised by its appearance. F. H. L.

Volumetric Estimation of Tannin. L. Specht and F. Lorenz. (*Chem. Zeit.*, 1900, xxiv., 170.)—This process depends on the precipitation of safranine as a tannin-antimony lake, using an excess of dye, and determining that excess by titration with a standardized hydrosulphite. The reagent is prepared by suspending 50 grammes of zinc dust in 100 c.c. of water, and adding, at a temperature not exceeding 36° C., 600 c.c. of a 20° Beaumé solution of ammonium bisulphite previously neutralized with ammonia. When the precipitate has settled, 75 c.c. of the clear supernatant liquor are diluted to 2 litres, and the solution is again clarified by subsidence. To standardize the hydrosulphite, it is titrated against (1) a weak aqueous solution of safranine "T extra," of the badische Anilin Fabrik, twice recrystallized from alcohol, and (2) against the same volume of water just tinted pink with the dye solution. From the figures so obtained are calculated the value of the hydrosulphite in terms of safranine, and the amount of reagent destroyed by the oxygen dissolved in the water. To carry out the analysis, about 0·16 gramme of tannin, 0·2 gramme of tartar emetic, and 0·33 gramme of safranine are dissolved separately, mixed together, diluted to 2 litres, and allowed to stand for five or six hours, with occasional agitation. A portion of the clear liquid is finally titrated with the hydrosulphite till the colour disappears. Deducting from the volume of reagent used the quantity decomposed by the oxygen dissolved in the water, the remainder indicates the amount of safranine unprecipitated; and thus the weight of dye in the lake, or the value of the tannin, may be deduced.

In order to express the latter in figures, parallel experiments with a pure tannin, or with a tannin of known degree of purity, must be conducted simultaneously. The titration should be done in a beaker under a layer of oil, the jet of the burette entering the aqueous liquid; and the hydrosulphite should be stored in a reservoir, connected as usual to the base of the burette, and also covered with oil. The reagent thus retains its strength practically unchanged for twelve hours. All the water employed should be thoroughly boiled. When using pure tannin as the standard, the substance should be air-dried merely, the proportion of moisture in it being determined in a separate portion. F. H. L.

Estimation of Uric Acid by Precipitation as Ammonium Urate. E. Wörner. (*Zeits. physiol. Chem.*, xxix., 70; through *Zeits. angew. Chem.*, 1900, 141.)—This process depends on Fischer's statement (*Ber.*, 1899, xxxii., 3266) that uric acid can be heated with dilute caustic soda without losing ammonia. 150 c.c. of urine are warmed to 40° or 45° C., and 30 grammes of ammonium chloride are dissolved in it. After standing for about an hour the precipitated ammonium urate is filtered off and washed with a 10 per cent. solution of ammonium sulphate till it no longer gives a chlorine reaction. The residue is dissolved in hot 1 or 2 per cent. caustic soda, and the paper is washed with hot water. The filtrate is then heated on the water-bath until all free ammonia has disappeared, and finally kjeldahled with 15 c.c. of strong sulphuric acid and a little copper sulphate. One c.c. of decinormal sulphuric acid corresponds to 0·0042 gramme of uric acid.

It seems improbable that other substances should be thrown down from the urine by this treatment. Small amounts of albumin are without effect; larger quantities are objectionable. Urines containing urate or phosphate deposits can be used directly. F. H. L.

INORGANIC ANALYSIS.

Lead Assay. J. Flath. (*Chem. Zeit.*, 1900, xxiv., 263.)—The author states that the process of determining lead in ores, residues, etc., by reduction under fluxes in a wrought-iron crucible is more accurate than is often imagined. He quotes results obtained with fourteen products found to contain by wet analysis from 1·25 to 76·15 per cent. of lead, which show a maximum deficiency of 1·48 (mean −0·79) per cent. in the yield calculated on the samples when treated by the fusion method. The best composition of the flux either for an acid or a basic material is sodium carbonate, 70 parts; borax, 28 parts; and potassium bitartrate, 2 parts; but the proportions need not be maintained exactly. F. H. L.

Determination of Thallium as Normal or Acid Sulphate. P. E. Browning. (*Zeits. anorg. Chem.*, 1900, xxiii., 155.)—Thallium can be determined as acid sulphate by heating its chloride with sulphuric acid to a temperature between 220° and 240° C., or as normal sulphate by igniting it similarly at a dull-red heat. Both processes are perfectly satisfactory. F. H. L.

The Rapid Evaluation of Metallic Tungsten Powders. F. Ibbotson and H. Brearley. (*Chemical News*, vol. lxxx., pp. 294, 295.)—The following method is more rapid than the usual one, and gives as near an approximation to the truth:

Three grammes of the substance are weighed in a tared platinum dish, then ignited to tungsten trioxide and weighed (A). After treating the mass with hydrogen fluoride it is re-weighed (B), the loss indicating silica. To the residue is added water and pure sodium hydroxide, and the whole is diluted to 200 to 300 c.c., boiled, filtered through paper pulp, ignited, and weighed (C), then treated with a little hydrochloric acid, diluted, filtered, and the precipitated trioxide ignited, and weighed (D).

B = tungsten trioxide and its iron and manganese compounds, which in turn are represented by C – D; therefore B – C + D = the amount of tungsten trioxide.

The residue D and the filtrate from the sodium hydroxide treatment contain the whole of the tungsten, which may be estimated direct if required; and the filtrate obtained previous to the final weighing (D) contains the iron and manganese.

Sulphur, if not removed during the hydrochloric acid treatment or in roasting, makes the results too high. It should not, however, be present in tungsten powders used for steel-making.

Carbon may be estimated by roasting in a current of oxygen, or by combustion with lead oxide. C. S.

The Action of Ammonia upon Magnesium Salts. W. Schieber. (*Oesterr. Chem. Zeit.*, 1900, iii., 83.)—According to the text-books, magnesium salts are not quantitatively precipitated by ammonia, while if sufficient ammonium salts are present no precipitate is formed at all. These phenomena are explained by the production of a double compound of the typical formula $(MgCl_4)(NH_4)_2$, which is not decomposed by ammonium hydroxide. It is noticeable that the authorities say nothing about the influence of temperature; these statements refer only to cold solutions; in hot liquids, even in presence of 4 molecules of an ammonium salt per 1 molecule of magnesium, sufficient ammonia will cause a turbidity, if not a precipitate. The matter is important in water analysis, where alumina is thrown down by boiling with ammonia, for the deposit may also contain magnesia, which is scarcely capable of re-solution under the working conditions. From a special series of experiments the author finds that to insure all magnesium remaining in solution in such separation it is necessary to have an excess of ammonium chloride, and to use as little ammonia as possible. Employing normal solutions of magnesium sulphate, ammonia, and ammonium chloride, the following ratios show the maximum proportion of ammonia and the minimum proportion of ammonium chloride which will allow the mixtures to be raised safely to the boiling-point:

$MgSO_4$	NH_3	NH_4Cl
1	2	1
1	10	2

F. H. L.

Composition of Ammonium Magnesium Arsenate. M. Austin. (*Zeits. anorg. Chem.*, 1900, xxiii., 146.)—This precipitate will be obtained of theoretical compo-

sition provided a faintly acid solution of arsenic acid, free from ammonium salts, is treated with 30 c.c. of ammoniacal magnesia mixture more than is necessary to throw down the arsenic, the total volume of liquid not being allowed to exceed 200 c.c. If the precipitate is filtered off and washed with 25 c.c. of ammoniacal water, no arsenic will remain in solution. Ammonium chloride has a slight solvent action upon the double arsenate; and although this effect can be overcome by employing a larger excess of magnesia mixture, the chloride also has a tendency to cause the displacement of part of the magnesium by ammonium in the original precipitate. The author's magnesia mixture contains 110 grammes of crystalline magnesium chloride and 58 grammes of ammonium chloride in 2 litres of water, to which are added 10 c.c. of "ammonia." F. H. L.

Phosphotungstic Acid as a Test for Potassium. **E. Wörner.** (*D. Pharm. Ges. Ber.*, 1900, x., 4; through *Chem. Zeit. Rep.*, 1900, 48.)—A 10 per cent. solution of ordinary crystalline phosphotungstic acid forms a more sensitive reagent for potassium than either platinic chloride or tartaric acid. From acid solutions the potassium salt falls in coarse crystals, from a neutral solution it separates in a very fine state of subdivision, rendering the liquid milky. Barium, strontium, calcium, and magnesium are not thrown down by the reagent; sodium chloride is only precipitated from a saturated solution, and the deposit dissolves immediately on dilution. Ammonium compounds must be previously removed. F. H. L.

Estimation of Iodic Acid in Sodium Nitrate. **R. Auzenat.** (*Monit. Scient.*, 1900, xiv., 72; through *Chem. Zeit. Rep.*, 1900, 48.)—Six similar test-tubes, 18 centimetres tall and 25 millimetres in diameter, each provided with a mark at the 50 c.c. point, are placed side by side in a stand in front of a white background. In the first are put 10 c.c. of a solution of potassium iodate (1 gramme per litre); in the second 30 c.c., in the third 35 c.c. . . . in the sixth 50 c.c. of a solution of the nitrate under examination (33 grammes per litre). The tubes are then filled up to the mark with water, and 2 c.c. of 10 per cent. potassium iodide and 5 drops of glacial acetic acid are quickly added to each. After shaking gently and standing for ten minutes the colours are compared with the standard tube (No. 1); but if none of them is a satisfactory match, the whole test is repeated with a different quantity of potassium iodate. Commercial sodium nitrate generally contains iodic acid equivalent to 1 or 2 per cent. of sodium iodate. F. H. L.

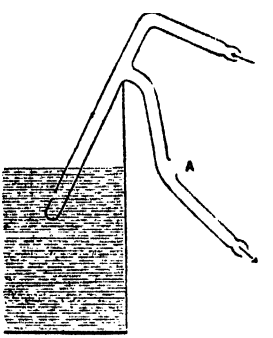
Determination of Phosphoric Acid as Phospho-Molybdic Anhydride. **J. Hanamann.** (*Zeits. landw. Versuchsw. Oesterr.*, 1900, iii., 53; through *Chem. Zeit. Rep.*, 1900, 72.)—The author strongly advocates the direct method of estimating phosphoric acid indicated by the above title. He employs the Wagner-Stutzer reagent: 150 grammes of ammonium molybdate and 400 grammes of ammonium nitrate dissolved in *two litres* of water and poured into 1 litre of 1·19 nitric acid. Twenty-five c.c. of the phosphate solution are mixed with 50 c.c. of the reagent,

warmed very slowly up to 40° C., and stirred for ten minutes; the precipitate is washed with acid ammonium nitrate, then with alcohol, and dried, ignited, and weighed. This ignition must be done uniformly, avoiding too great heat. His factor for the conversion into P_2O_5 is 0·03946. F. H. L.

Erdmann's Reagent [Amidonaphthol-K-Sulphonic Acid] for Nitrites in Water. H. Mennicke. (*Zeits. angew. Chem.*, 1900, 235.)—The present author agrees that Erdmann's new reagent (this vol., p. 81) is most useful. Its indications are perfectly certain. They are not affected by the presence of ferric chloride, nitric acid, or other oxidizing agents; a large excess of the latter bodies only causes the colour to be more wine-red. The limit of sensitiveness for a sharp eye in daylight is 1 part of sodium nitrite per 300 million—*i.e.*, it is about 300 times more delicate than starch and potassium iodide. The colour attains its maximum intensity in half an hour. Mennicke quotes analyses of several town and waste waters which fully bear out his statements. F. H. L.

APPARATUS.

New Form of Water-bath Regulator. H. S. Hatfield. (*Chemical News*, vol. lxxxi., p. 65.)—To obviate the stoppage of the supply by accumulations of air in the ordinary syphon-tube regulator, the author employs the form illustrated in the drawing. The supply-tube is bifurcated, one limb dipping under the liquid of the bath, and the other being open to the air at A. The water in the bath will rise only slightly above the level of A, the excess being due to the force necessary to divert the stream. All the tubes must be full of water; any air-bubbles formed are swept away by the current, which must be strong enough for this purpose. The water must leave the sides of the glass at A.

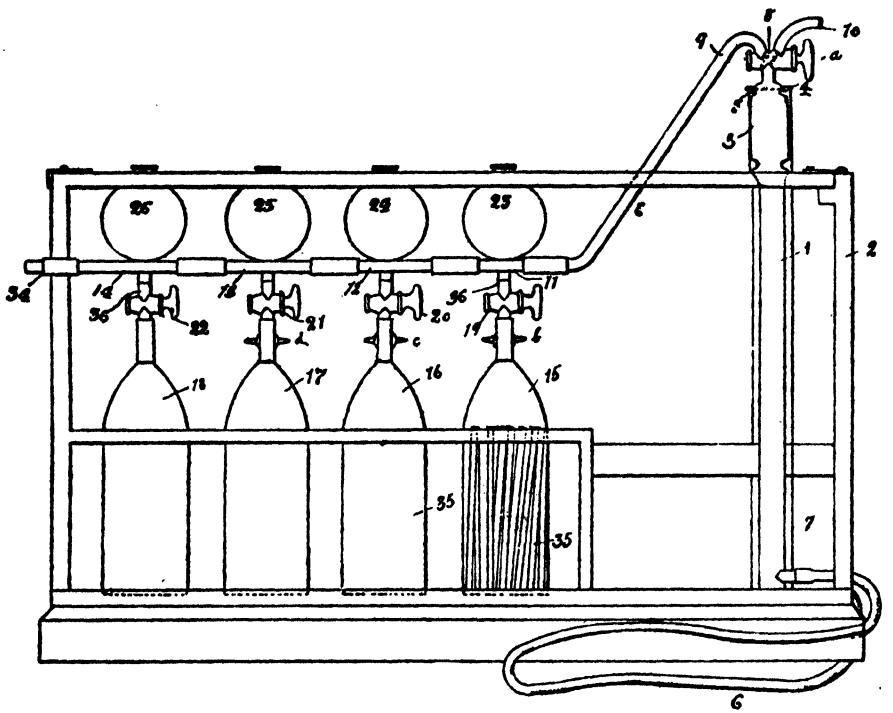


C. S.

Apparatus for the Analysis of Illuminating and Fuel Gases. G. E. Thomas. (*Journ. Amer. Chem. Soc.*, vol. xxi. [12], pp. 1108-1112.)—The apparatus measures 16 inches long by 15 inches high. The burette, 1, is graduated to 100 c.c., 40 of which (the bulb, 3) are in whole divisions, and the rest in 0·2 c.c. It is fitted with platinum terminals, 4, 5, and filled with water acidified with sulphuric acid to decrease the solubility of the carbon dioxide. The absorption bottles, 15, 16, 17, and the storage bottle, 18, are connected with the burette by a capillary tube, *e*, and the T tubes shown, these latter being provided with rubber connections. Nos. 15 to 17 are fitted with stop-cocks, *b*, *c*, *d*. Bottle 15 is filled with caustic potash solution, the surface being increased by the use of glass tubes; No. 16 contains bromine water, and No. 17 (protected from light) phosphorus under water.

The burette and capillary tube, 10, being filled with water by raising the levelling

tube, 7, the tap, *a* is closed, 10 is connected with the gas supply, and 100 c.c. of gas are drawn into the burette by lowering 7 and reopening *a*. The gas is then forced into bottle 15, the operation being thrice repeated, and the loss (carbon dioxide) in volume measured after draining. The illuminants and oxygen are then measured by absorption in bottles 16 and 17 respectively, care being taken to absorb the bromine vapours in the gas by bottle 15 before measuring the residual gas. The unabsorbed remainder is collected in 18 for explosion tests. For these latter about 80 c.c. of air are drawn into the burette through 10, followed by about 15 c.c. of gas from 18, water being drawn through the capillary as far as *a* from bottle 17. The mixed volume having



been carefully measured, mixing is completed by passing the air and gas into 15 and back again. The tap, *a*, is closed and the pressure reduced by lowering 7; and a water-seal is formed by bending the rubber tube and securing it in a slit in the frame. The spark is then passed, the explosion being very mild owing to the peculiar construction of the burette; and, when cooled down, the contraction in the volume, the amount of carbon dioxide, and the excess of oxygen are determined successively, the water from the burette being finally forced through the capillary system into 17, to insure complete absorption. The final volume contains only the nitrogen in the air plus that in the gas.

For fuel-gas analysis 17 and 18 are charged with cuprous chloride solution, air being excluded by a seal of petroleum in the funnels. A lubricant is necessary to insure success, vaseline being suitable for the bromine bottle. C. S.

THE ANALYST.

JULY, 1900.

PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS.

THE monthly meeting of the Society was held on Wednesday evening, June 6, in the Chemical Society's Rooms, Burlington House. The President (Mr. W. W. Fisher, M.A.) occupied the chair.

The minutes of the previous meeting were read and confirmed.

A certificate of proposal for election to membership in favour of Mr. John Stewart Remington, analytical and consulting chemist, Corporation Street, Lancaster, was read for the first time.

Messrs. J. R. Brooke, T. H. Lloyd, and G. H. Morris, Ph.D., were elected members of the Society.

The following papers were read: "The Determination of Oxygen in Copper by Ignition in Hydrogen," by L. Archbutt; "Uniformity in the Conduct of Soil Analysis," by A. D. Hall, M.A.; "The Adulteration of Wheaten Flour with Maize," by G. Embrey; and "A New Colour Reaction for Distinguishing between certain Isomeric Allyl and Propenyl Phenols," by Alfred C. Chapman.

THE INFLUENCE OF TEMPERATURE AND CONCENTRATION ON THE SALINE CONSTITUENTS OF BOILER WATER.

BY CECIL H. CRIBB, B.Sc., F.I.C.

(*Read at the Meeting, April* 4, 1900.)

THE behaviour of the saline constituents of water when used in high-pressure boilers has, as far as I am aware, never formed the subject of a paper before this society. The matter has only a remote connection with analytical work, but to all who are likely to be consulted on questions relating to boiler waters it should be of interest.

Personally, I have not unfrequently been asked by engineers what would happen to a water when heated in a boiler working at a certain pressure, or what proportion of its dissolved salts it would deposit. On looking through the literature of the subject, I was surprised to find how little really definite information was to be had.

The common use of high-pressure feed-water heaters shows that the effect of pressure, *i.e.*, temperature, on the solubility of the salts is fully recognised and taken

advantage of; but I know of no systematic investigation such as would enable one to predict with even approximate certainty the changes in composition that would be brought about when a water is heated above 100° C., or the further changes due to the concentration which necessarily occurs in a boiler working under the ordinary conditions. Again, as analysts, we are frequently called upon to say whether, judging from analysis, a water will, or will not, have any action on the boiler or its fittings. In most cases the problem is a fairly simple one, but in some instances a little knowledge as to what a water is likely to become after concentration to from five to twenty times its original strength might be of considerable use. Even with the best of boiler waters a certain amount of corrosion takes place, and it is not unreasonable to suppose that, in this connection, the concentration is not without influence. In any case, the high temperatures have a marked effect on the chemical activity of those constituents to which corrosion is usually attributed.

The problems to be dealt with are somewhat complicated, because it is impossible, with steam-boilers working under ordinary conditions, to separate the influence of pressure from that of temperature, and these, again, from that of the concentration of the more soluble constituents. The effects of all three are, moreover, obscured by the constant addition of fresh feed water, diluting the boiler contents, and at the same time bringing with it fresh supplies of saline matters, some of which add to the concentration, while others are speedily deposited because of the sudden increase of temperature to which they are exposed, or because of the superabundance of certain salts in the surrounding medium.

The various analyses under the heading "Water from Boiler" (Tables I., III., IV., V., VI. and VIII.) show the sort of samples that are obtained from boilers which have been running for various periods up to six or seven weeks. Of course, in the rare instances in which pure sea-water is used in high-pressure boilers, the degrees of salinity here shown are enormously exceeded, but I have not been able hitherto to obtain any specimens of this sort for examination.

In Table I. is given the composition of a very bad feed water, and of a sample taken from a water-tube boiler in which it had been used for some weeks. The analyses are far from complete, but a brief glance at them will serve as an introduction to the more complicated series of analyses that follow. In this and all the other analyses of waters the results are expressed in parts per 100,000:

TABLE I.

	Feed Water.	Water from Boiler.	Concentration Factor.
	301·5	1,601·2	5·31
	145·5	670·0	4·64
Total hardness:	95·2	183·1	1·91
Permanent hardness:	54·4	183·1	3·34
Temporary hardness:	40·8		

It will be seen at once that all the constituents have become concentrated to a different extent. The numbers under the heading "Concentration Factor" were obtained by dividing the proportion of each constituent of the boiler water by that of the same constituent in the feed water, and they really represent the number of

volumes of the feed water that would contain the same amount of that particular constituent as one volume of the boiler water. If owing to the evaporation taking place in the boiler all the constituents had been concentrated to a like extent, all the concentration factors should be the same. They all, as would be expected, differ, and the question at once arises, Which of them, if any, indicates the real degree of concentration? Or, to put it in another way, have any of the constituents merely gone on accumulating in the boiler? or have they all suffered a certain amount of deposition in the solid form, or have they been decomposed? Obviously, the extent to which any particular salt has gone out of solution, owing either to deposition or decomposition, can only be ascertained if the real degree of concentration is known.

This can plainly only be got at indirectly, but some means of doing it is absolutely essential, so I propose to devote a short time to the discussion of the most suitable plan before proceeding further. In a laboratory experiment the actual solid deposit could be collected, weighed and analysed, or the total amount of water actually used could be measured and compared with the volume of the residual water, but obviously such methods are out of the question in the case of boilers working under ordinary conditions. If, however, one constituent can be found which never goes out of solution, or leaves the water in the boiler owing to decomposition of any sort, then that will serve as a direct measure of the concentration, making it possible, if the capacity of the boiler is known, to calculate the actual volume of water evaporated, and consequently the amounts of each of the other constituents which should remain in the concentrated water had they, too, suffered no loss.

The chlorides, the nitrates and the alkalies are on the whole the most likely to answer the purpose. The nitrates are, however, likely to become reduced, either by organic matter or the metal of the boiler, and the estimation of the alkalies is troublesome and not too accurate, so that one naturally turns to the chlorides. All the chlorides are so soluble in water that they are never likely to come out of solution from mere concentration alone except under the most extraordinary circumstances. Hence it is rare to find scales and deposits from land boilers containing any considerable proportion of chlorine, and, as a rule, that element is present only in minute traces. According to Vivian Lewes ("Service Chemistry," p. 119), even sea-water when evaporated does not begin to deposit salt until about 24 per cent. of the latter is present in solution; and the same author found an incrustation from sea-water to contain only 2·79 per cent. of sodium chloride. In my own experience I have found this figure to be very much exceeded. Thus, a scale from a naval water-tube boiler of recent construction, in which, owing to an accident, much sea-water had to be used, had the following composition, showing that it consisted almost entirely of sodium chloride:

TABLE II.

	Per Cent.
Fe_2O_3	0·28
CaO	0·19
MgO	1·40
SO_3	1·72
Cl	58·49
OH_2...	0·81

No ordinary water is ever allowed to concentrate in a boiler to such an extent as to even approach sea-water in salinity, so that the chances of the salt crystallizing out can be safely neglected. On the other hand, the presence of magnesium chloride has to be reckoned with. There seems to be a good deal of doubt, judging from text-books, as to the behaviour of this substance when heated.

Some, *e.g.*, Bloxam, state definitely that solutions of magnesium chloride decompose on evaporation, giving off hydrochloric acid. Comey's "Dictionary of Solubilities," Watts' "Dictionary of Chemistry" (Morley and Muir), and Blount and Bloxam's "Engineering Chemistry" say practically the same, while most of the other English text-books refer only to the behaviour of the solid substance. There is, in any case, a widespread impression amongst chemists that the solution is decomposed on heating at ordinary pressures.

Those text-books that mention the decomposition of the solution plainly do not refer to evaporation under pressure, and the original fountain-head of their information seems to be a paper by Casaseca (*Comptes Rendus*, 1853, xxxvii., 350), who found that a solution of magnesium chloride, when heated, begins to decompose when it contains only six molecules of water to one of the chloride. From the way he conducted his experiment, he was obviously *igniting* the ordinary crystalline form of the salt ($MgCl_2,6OH_2$).

When a solution of magnesium chloride is evaporated on a water-bath, I find that this salt, with six molecules of water, always remains behind, and the evaporation may be repeated any number of times without any decomposition occurring, the total solid residue remaining quite constant, and always dissolving without difficulty to a clear solution. The proportions of chlorine and of magnesium also show no variation.

If the salt be melted in its own water of crystallization, but little visible decomposition takes place until a temperature of about 150° C. is reached, and the thermometer rises slowly to about 180° C. There can be no doubt that the general impression as to the instability of solutions of magnesium chloride when heated is due to the misleading employment of the term "evaporation" in the text-books.

I find, further, that a solution of magnesium chloride, so strong that it deposited crystals of the hydrated salt in the cold, may be distilled to further concentration without giving off a trace of acid. The same experiment repeated, with the addition of a large quantity of iron filings to the solution, gave a like result, which seems to show that the metal of the boiler would be without influence in this connection. Vivian Lewes ("Service Chemistry" p. 121), states that "when sea-water is evaporated in contact with a large surface of metallic iron, no chloride can be detected in the distillate until four-fifths of the solution has distilled over." Such a concentration of sea-water would not contain more than $2\frac{1}{2}$ per cent. of $MgCl_2$, while the solution I employed was nearer 50 per cent., so that further investigation of the matter is required. The behaviour of magnesium chloride solutions when heated under pressure still remains to be considered. That the salt, if present in the feed water, is almost entirely decomposed in the boiler is plain from the fact that all the analyses in the tables (IV., V., VI. and VIII.) show a remarkable disappearance of the MgO from the water after it has entered the boiler. Magnesium hydroxide is one of the commonest constituents of boiler incrustations, while chlorine in

any quantity is rare. This holds good even when there is much Mg and Cl in the feed water, and is very strikingly shown by the analyses in Table III., in which the feed water contains very large proportions of both MgO and Cl, while the scale is practically free from chlorine, but contains nearly one-third of its weight of magnesia :

TABLE III.

	Feed Water.	Scale from Boiler.
Total solids	938·5	
Chlorine (Cl)	460·0	Moisture trace
Carbonic acid (CO_2) ...	13·38	0 per cent.
Sulphuric acid (SO_3) ...	45·76	39·87 ,,
Lime (CaO)	117·88	28·00 ,,
Magnesia (MgO) ...	129·84	30·14 ,,
Nitric acid (N_2O_5) ...	9·55	Insoluble 3·19 ,,

The magnesium has therefore plainly left the water under the influence of the high temperature to which it has been exposed. What has happened to the chlorine? Unfortunately, in this particular case, I have no analysis of the water after it had been in the boiler, but it by no means follows that because magnesium chloride is decomposed and its base deposited that the chlorine leaves the water at all. The process may be the result of a double decomposition with some other salts; but even if free hydrochloric acid is formed, it is far from certain that it would be evolved as gas and pass away with the steam. That it does not do so in the case of waters reasonably suited for steam-raising is clearly shown by Table IV., which gives the composition of the contents of a boiler using a London water after running for about six or seven weeks, and that of the condensed water collected after the steam had passed through a superheater :

TABLE IV.

Lancashire Boiler : 40 lb. pressure.

	Water from Boiler.	Condensed Steam.
Total solids	503·2	2·60
Chlorine	94·0	0·21
Nitric acid (N_2O_5)	2·65	0·016
Carbonic acid (CO_2)	5·98	0·14
Sulphuric acid (SO_3)	127·68	0·49
Silica (SiO_2)	1·92	0·20
Oxide of iron (Fe_2O_3)	0·72	0·16
Lime (CaO)	18·24	0·65
Magnesia (MgO)	0·33	0·15

In view of the concentration, a good deal of magnesium must have been deposited in the form of sediment or scale, and yet the condensed water shows no excess of chlorine, but rather the reverse. It is, in fact, obvious in the case of ordinary waters that no hydrochloric acid passes away in the steam, as otherwise the engines would *always* be attacked. Further, it has been recently shown by Bailey and Johnston (*J. S. C. I.*, 1899, p. 455) that an aqueous solution of hydrochloric acid does not give off any acid when distilled until the proportion of the latter reaches nearly 1 per cent.

Of course, under a high pressure something entirely different may happen. Table V. which relates to a water which contained a fair proportion of MgO and much chlorine, and acted powerfully on the boiler, and its fittings* affords an indirect proof that no appreciable amount of chlorine leaves the boiler:

TABLE V.

Pressure: 140 *lb.*

	Feed Water.	Water from Boiler.	Concentration Factor.
Total solids	99·2	411·0	
Nitric acid (N_2O_5)	Trace	Trace	
Chlorine (Cl)	18·15	93·5	5·15
Sulphuric acid (SO_3)	16·76	84·8	5·06
Carbonic acid (CO_2)	15·21	38·94	
Silica (SiO_2)	1·30	7·0	
Oxide of iron, etc.	0·40		
Lime (CaO)	7·77		
Magnesia (MgO)	3·33		
Na_2O, and K_2O	44·89	209·9	4·68

The water was strongly alkaline owing to the presence of sodium carbonate, and it is not unreasonable to suppose that the lime and magnesia, which are completely absent from the water after heating, were deposited in the form of carbonates. In the absence of lime and magnesia, there is no more reason for the SO_3 leaving the water in the boiler than for the mixed alkalies or the chlorine, consequently the concentration factors for these three should be identical within the limits of experimental error. This is the case with two of them, and the third is not far out. In any case, the figure for the Cl is the *highest*, as, indeed, it almost invariably is throughout the tables. In this particular analysis, at all events, the amount of chlorine is almost certainly a true measure of the concentration; in the others it is, if not the true one, at any rate the best available.

In the present connection, as well as from a theoretical standpoint, the behaviour of magnesium chloride when its solutions are heated under pressure is of such great interest that I have commenced its investigation, and hope to publish the results elsewhere. I may say briefly that under a pressure of one atmosphere (temperature about 125° C.) practically nothing happens to a solution containing 2·25 per cent. of $MgCl_2$, but at five atmospheres a solution of the same strength is partially decomposed, with deposition of MgO and evolution of HCl, the extent of the decomposition being determined by the duration of the experiment and the freedom with which the steam is allowed to escape. Working with the pure salt, however, the conditions are entirely different to those which obtain in a boiler in which a water containing a mixture of many salts is being evaporated, and the large surface of iron exposed to the liquid may profoundly affect the result.

In any case, I propose to assume, in the absence of satisfactory evidence to the contrary, that in the case of the waters referred to in Tables IV., V., VI. and VIII. the chlorine is a sufficiently exact measure of the concentration to render it possible

* This boiler worked at 140 pounds pressure; other boilers with the same water, working at 80 pounds, gave no trouble.

to calculate the original volume of feed water used. The use I propose to make of the concentration factor obtained in this way will appear later.

Table X. (p. 181) shows the composition, apart from the alkalies, oxide of iron, and silica, etc., of four waters: (1) New River water; (2) water from a boiler for which it was used, after working 82 hours at from 70 to 80 pounds pressure; (3) after working 468 hours at the same pressure; and the same water evaporated at a pressure of only 6 ounces per square inch till the chlorine contents reached that in No. 3.

Table VI. deals with a still more extended series of samples, for which I am indebted to Messrs. W. H. Allen, Son, and Co., of Bedford. The details as to the samples are stated in the tables; the intervals at which the samples were taken are complete days, whereas in Table X. they are hours of actual working.

To get some idea of the effect of concentration alone, Table VI. may be studied in more detail.

TABLE VI.

Bedford water, 180 pounds pressure; boiler evaporates 3,000 gallons in 10 hours; samples collected at intervals of 1, 8, and 24 days from date of lighting up.

	From Hot Well.	After 1 Day.	After 7 Days.	After 24 Days.
Total solid matter ...	33·0	106·12	171·0	341·7
Chlorine (Cl) ...	2·85	14·95	41·15	90·0
Carbonic acid (CO_2)	1·27	0·97	1·22	3·50
Sulphuric acid (SO_3)	11·36	41·02	49·58	82·52
Lime (CaO)	7·35	8·00	5·34	9·20
Magnesia (MgO) ...	1·35	2·77	0·47	0

As would of course be expected, the water at the end of twenty-four days has become highly concentrated, but the results differ for each constituent. Thus, while the total solids in twenty-four days has reached ten times its initial amount, the concentration of the carbonic anhydride is only 3 times, that of the chlorine 30 times, the sulphuric acid 7 times, the lime 1·2, and the magnesia has disappeared altogether. Of course, any comparison between the feed-water and any of the other samples necessarily takes into account the influence of the high pressure and temperature to which the latter have been exposed. But if any one constituent is taken, and the effect of time alone is studied, the figures still require a good deal of interpretation. Thus, the chlorine is 5 times as great after working one day as it was before entering the boiler, but six further days' work only produce 3 times the effect of the one day, and twenty-three days' more only 6 times. The SO_3, again, is 3 times as great after one day, 4·3 times after seven days, and only 7 times after twenty-four days. In fact, of all the constituents estimated not two behave alike.

A very little reflection will, however, be enough to explain some of these seeming contradictions. Thus, the apparent effect of the initial day of working will always be greater than that of any subsequent one (as far, at all events, as the CO_2, SO_3, lime, and magnesia are concerned), because the boiler is started with its contents undiluted with condensed water from the engine.

That the changes produced in a unit of time should decrease as the concentration increases is what would naturally be expected, in view of the fact that the solid matter entering the boiler with the feed water must bear a steadily decreasing ratio

to the ever-growing amount of dissolved matter already there. The ratio will, of course, be partly dependent on the proportion existing between the evaporative power and the capacity, at working level, of the boiler—a proportion which varies very widely in the different types of boiler.

That the individual constituents differ from one another in the extent to which they have become concentrated is, of course, owing to the greater insolubility of some of them at high temperatures, or to their insolubility in strong solutions of the other saline constituents, of which by far the most important in this, as in the majority of waters, is the sodium chloride. Further changes may also be produced by double decompositions, giving rise to less soluble compounds.

A very cursory inspection is sufficient to show that there is a continual falling out of solution of the majority of the salts, but the full extent of this is marked by the mere accumulation of what remains. To get any idea of how much has gone out of solution, it is necessary to consider the amount of feed water that the contents of the boiler at any time represent, *i.e.*, to know the true concentration factor. I have already given reasons for assuming that the chlorine is the best guide to this.

On this assumption it is only necessary to divide the chlorine in the boiler water at any period by the chlorine in the feed water to find out how many volumes of the latter one volume of the water in the boiler represents, this figure being, of course, the true concentration factor.

If this figure in turn be divided into the amount of any constituent, it will give the proportion of that constituent still remaining dissolved in the boiler water expressed in parts per 100,000 of the *feed water*.

Thus, taking the chlorine in Table VI., the amount after one day, viz., 14·95, divided by 2·85 (the amount in the feed water), gives 5·2; this is the concentration factor, and indicates that the water after one day in the boiler contains Cl belonging to 5·2 times its volume of feed water, and, if the assumption already referred to be correct, not only the chlorine, but each of the other constituents as well, unless the actual amount has been reduced by the particular consituent leaving the water owing to deposition as scale or sludge, or by evolution in the gaseous form. If 5·2 be now divided into 14·95, the amount of Cl belonging to one volume of feed water is obtained, *i.e.*, 2·85, the same as in the feed water, because, according to the initial assumption, no chlorine is lost. If 5·2 be divided into the other figures for the same period of working, the results differ from the proportions of the same constituent in the feed water, because all the constituents, other than the chlorine, have suffered loss either by deposition as sediment or scale or by evolution as gas.

TABLE VII.

After compensating for Water evaporated.

	From Hot Well.	After 1 Day.	After 7 Days.	After 24 Days.
Total solid matter ...	33·0	20·23	11·84	10·56
Chlorine	2·85	2·85	2·85	2·85
Carbonic acid	1·27	0·184	0·085	0·108
Sulphuric acid	11·36	7·82	3·43	2·55
Lime	7·35	1·525	0·396	0·285
Magnesia	1·35	0·528	0·032	0

Table VII. is obtained in this way, viz., by dividing the respective concentration factors for each sample, calculated from the chlorine, into all the other figures for the same sample. The effect is practically to restore the water that has been evaporated, thus doing away with the merely cumulative effect of the concentration, and laying bare the combined effect of the high pressure and temperature and of the increasing salinity on the solubility of the various salts.

The total solid matter obviously suffers from any changes that affect its constituents. In Table VI. it has increased to about ten times its original amount, but Table VII. shows that as long as the experiment lasted solid matters continued to leave the water, and at the end of the twenty-four days $33{\cdot}0 - 10{\cdot}56 = 22{\cdot}44$ parts of solid matter per 100,000 of feed water used had actually disappeared.

The carbonic acid, owing to the use of a hot well and of condensed water, is very small in the feed water, and the subsequent differences in Table V. hardly exceed the ordinary error of experiment, except in the case of the last. This sudden increase I am not prepared to explain. It may mean that calcium carbonate is more soluble in the presence of much sodium chloride, or it may be the result of some double decomposition between the already deposited carbonate and the salts in solution. In any case, the difference is not very great.

The sulphuric acid in Table VI. steadily increases, but with no apparent relation to the time. Table VII. shows that there has been a steady and continuous loss of SO_3 from the solution.

The lime, to which, with the magnesia, the greatest interest attaches, is apparently slightly more after one day in the boiler than in the feed water; but when allowance is made for the concentration, it is apparent (Table VII.) that that constituent has suffered more heavily than all the others, only one-fifth of the total amount present in the feed water remaining at the end of one day. The temptation to speculate as to its fate is very great, and I much regret that the analyses are not complete. They were made some time ago, when I did not realize the importance that might attach to the alkalies. The amounts of lime that have vanished, calculated from Table VII., seem to bear no regular relation to the CO_2 and SO_3 which have disappeared, and it seems probable that double decompositions between the scale and sediment and the dissolved salts are constantly occurring. In any case, it would be unjustifiable to base any theories on such a small number of analyses.

As there is always far more than an equivalent amount of SO_3, it is fair to regard the lime left at the end of twenty-four days as being all present as sulphate; the amount found, viz., 9·20, is equivalent to 22·34 parts of calcium sulphate.

Tilden and Shenstone (*Phil. Trans.*, part 1, 1884, p. 23) found that 100,000 of pure water at a temperature which gave a pressure of 132 pounds per square inch held in solution 27·0 parts of calcium sulphate, and at 513·5 pounds only 18·0 parts, and, calculating from these figures, I find that at 180 pounds 26 parts per 100,000 should remain in solution, so that it seems probable that the point of saturation for calcium sulphate at this temperature had been reached. Tilden and Shenstone's figures were obtained with pure water, so that it is plain that the concentration of the salts in the boiler water has not materially affected the solubility of the calcium sulphate. In Table X., column 3, however, although the concentration has not been

carried nearly as far, the sulphate of lime, calculated from the CaO, cannot exceed and is almost certainly less than 42·6 parts per 100,000, whereas Tilden and Shenstone found 56 parts to be soluble at that pressure. Here, therefore, it is quite plain that the saturation-point for 80 pounds pressure is not nearly reached, and yet it is obvious from Table XI. that SO_3 in some form has been continuously deposited since the boiler commenced to work. So, too, in Table IV. the lime is equivalent to 44·29 parts of calcium sulphate, while, according to Tilden, about 53 parts should be soluble.

To return to Tables VI. and VII. The magnesium exhibits similar peculiarities of behaviour to the lime, but it is very interesting to note that at the end of twenty-four days it has absolutely disappeared.

Although in Table VI. the quantity shows an increase at the end of one day, Table VII. makes it plain that a continual deposition of MgO in some form went on throughout the whole period dealt with.

In Table IV., which deals with a longer period, the MgO also nearly, but not quite, vanishes; and also in Table X.

In Table V. the disappearance is absolute, owing no doubt to the large proportion of alkaline carbonates present.

So far the tables have dealt with cases in which the pressure remained constant while evaporation went on. Through the kindness of Messrs. Marshall and Co., of Gainsborough, I was enabled to obtain samples from a boiler in which the reverse conditions obtained—*i.e.*, the pressure varied, and loss by evaporation was as far as possible avoided, thus affording an opportunity of studying the effect of pressure and the resulting high temperature, comparatively free from the disturbing influence of the concentration.

It is, of course, impossible in the case of steam boilers to separate the effect of pressure from that of temperature, as the one is the necessary concomitant of the other. But though water is commonly regarded as incompressible, the effects of pressure upon its volume are quite enough to produce important and far-reaching consequences by depressing the level of the ocean (Tait, *Challenger* Reports, "Physics and Chemistry," vii.). The pressures referred to here are, however, infinitely greater than anything occurring in boilers.

It is, I believe, generally admitted that hydraulic engineers are not troubled in any way by chemical changes in the water constituents brought about by the highest pressures they employ. Nevertheless, the possibility of the pressure itself playing some part in the changes taking place in the boiler contents must not be entirely disregarded, although everything seems to show that it can only be slight.

From a purely scientific point of view, the effect of pressure has been studied by Bunsen (*Ann. d. Chem. u. Pharm.*, 1848, 76-85), who obtained entirely negative results.

Sorby (*Proc. Roy. Soc.*, xii., 54) pointed out that Bunsen had failed to allow enough time for the effects to manifest themselves, and showed, by sealing up various substances in tubes which were completely filled with the cooled solution and warming them, that chemical changes followed by increase of volume are resisted, and those resulting in a decrease of volume are promoted, by pressure. Similarly

with solution: substances which expanded on entering into solution became less soluble under pressure, and substances which contracted became more soluble. The vast majority of substances contract, and therefore pressure increases their solubility.

The pressures he employed were, however, from 100 to 200 atmospheres, but even then the effects were extremely minute; for instance, the solubility of sodium chloride was only increased to the extent of 0·419 per cent. by a pressure of 100 atmospheres, that of potassium sulphate to the extent of 2·914 per cent.

The experiments of Tilden and Shenstone have already been referred to. Spring, of Liège (*Zeit. für Physik. u. Chem.*, 1889), found that when a diminution of volume accompanies solution, the presence of water facilitates the solidification of powders by pressure; and it seems extremely probable that in this direction, if at all, the pressure, apart from the temperature, affects the behaviour of the salts in boiler waters.

There is one important difference between the conditions which obtain in a boiler and those under which all the above investigations were made—namely, that in the former case steam in large quantity is continuously leaving the boiler, and is free to carry with it any gaseous products that may be formed.

TABLE VIII.

Gainsborough Water, heated up and kept for ten minutes at pressure of 50 lb., 100 lb., 150 lb., 200 lb., and 250 lb. per square inch. Steam kept as far as possible from escaping.

Temperature	138° C.	164° C.	181° C.	194° C.	205° C.	
Pressure	50 lb.	100 lb.	150 lb.	200 lb.	250 lb.	
	Feed Water.					
Total solids	58·4	58·6	54·10	54·52	52·88	43·29
Nitric acid (N_2O_5)	trace	trace	trace	trace	trace	trace
Chlorine (Cl)...	2·65	4·3	4·0	3·9	3·55	4·3
Sulphuric acid (SO_3) ...	20·06	23·28	23·48	23·77	24·11	16·88
Carbonic acid (CO_2)... ...	7·31	3·04	1·65	1·41	1·52	1·54
Silica (SiO_2)	1·10	0·18	0·83	0·45	0·46	0·42
Oxide of iron, etc.	0·30	1·03	0·18	0·30	0·11	0·15
Lime (CaO)	13·47	10·47	9·80	11·30	13·7	10·00
Magnesia (MgO)	6·29	7·00	5·74	4·41	2·73	1·81

TABLE IX.

After allowing for Water Evaporated.

Total solids	58·4	36·11	32·97	37·05	39·47	26·72
Nitric acid	trace	trace	trace	trace	trace	trace
Chlorine	2·65	2·65	2·65	2·65	2·65	2·65
Sulphuric acid	20·06	14·34	15·55	16·15	18·00	10·40
Carbonic acid	7·31	1·87	1·09	0·96	1·13	0·83
Silica	1·10	0·11	0·55	0·31	0·34	0·25
Oxide of iron	0·30	0·63	0·12	0·20	0·08	0·09
Lime	13·47	6·45	6·48	7·68	10·23	6·16
Magnesia	6·29	4·31	3·80	2·99	2·03	1·11

The samples dealt with in Table VIII. were drawn from a new boiler under the conditions stated in the table. The water was kept for ten minutes at the pressure

stated, and at the end of that time a sample was taken. The steam was kept as far as possible from evaporating, but it is quite evident that before 50 pounds' pressure was reached a considerable amount of concentration had taken place.

After that there are curious differences in the chlorine figures, for which I am quite at a loss to account, except on the supposition that some fresh feed water was allowed to get into the boiler. The time during which each of the stated pressures was maintained was unfortunately so short that the full effect could not be produced, and could the period have been twenty times as long it would have been much better. This, however, would have necessitated extending the whole experiment beyond one day, and could not conveniently be arranged for. The mistake, however, carried with it a certain compensation, for it disclosed a phenomenon which under other circumstances would probably not have been revealed. As a rule, water drawn from a boiler is, of course, very turbid, but the particles are large and settle down very quickly, leaving the supernatant liquid quite clear. In the case of those samples the water was turbid, or it would be better described as opalescent, but the particles were so fine that they were invisible when examined under the microscope with a one-sixth objective. It was absolutely impossible to filter the liquids clear, and I had to make the analysis with the turbid fluid. At the end of several months, however, the particles seemed to have got larger and heavier, and eventually sank to the bottom. When shaken up after this the liquid settled down clear almost immediately.

Dealing first with Table VIII., the total solids appear to be almost unaffected by 50 pounds pressure; but when allowance is made for the small amount of concentration that has taken place (Table VIII.), it is seen that there has been a considerable reduction, amounting in all to 22 parts per 100,000, of feed water, which appear to be made up, roughly speaking, of 5·7 SO_3, 5·4 CO_2, 7 of CaO, 2 of MgO, and 1 of SiO_2, while the Fe_2O_3 has increased, owing no doubt to action on the metal of the boiler.

At all the higher pressures the total solids undergo further diminution, with the exception of the figure in the fifth column, where there is a slight, but unmistakable, increase, which is more conspicuous when calculated on the feed water as in Table VIII. Between the time when a pressure of 150 pounds was reached and that when 200 pounds was registered, something seems to have happened which I cannot at present satisfactorily explain. The result is such as might be produced by a sudden inrush of feed water. In any case, I cannot attribute it to errors in the analytical determinations, as most of the results relating to these two samples were done in duplicate.

Between 200 and 250 pounds there is a sudden and very marked fall in the total solids, which may be fairly put down to the action of the increasing pressure and temperature on the solubility of calcium and magnesium sulphates, as it is plain from Table VIII. that the great bulk of the loss in total solids in the sixth column is accounted for by reductions in the SO_3, CaO, and MgO.

The lime is equivalent to 24·2 parts per 100,000 of calcium sulphate. Tilden and Shenstone's curve for the solubility of calcium sulphate in pure water indicates that from 25 to 26 parts per 100,000 should be soluble at the temperature attained under

the 250 pounds pressure, which is a sufficiently good agreement. At 200 pounds pressure, Tilden and Shenstone's curve gives the solubility as about 29 parts per 100,000, while the lime in the boiler water at that pressure is equivalent to 33 parts per 100,000 of calcium sulphate.

At the lower pressure, given in Table VIII., the point of saturation for calcium sulphate is never reached, and so the SO_3 remains practically the same or increases slightly; the lime decreases at first until most of the carbonate has been deposited—*i.e.*, till 100 pounds pressure is reached—after which it increases very slowly, until at 200 pounds a saturated solution remains.

The magnesium behaves in a somewhat unexpected manner. A considerable proportion of it remains in solution throughout the whole series of samples. In Table VIII., at 50 pounds pressure, there is actually more in solution than in the feed water, and, even when allowance is made for the concentration, the loss only amounts to one-third of the whole. It seems, therefore, fairly obvious that the carbonic acid which has gone out of solution is mainly combined with lime, unless, indeed, magnesium carbonate is deposited only to part with its carbonic acid immediately afterwards; but even then it is not easy to see how the latter could get once more into combination with any of the bases still left in the water.

From the time 100 pounds pressure is reached, practically no combined carbonic acid leaves the water, and yet the MgO goes on disappearing in regularly increasing amounts for every 50 pounds rise of pressure.

That the MgO should not be deposited as long as any CaO remains in solution is quite in accordance with the observations of C. H. Bothamley (*Chem. Soc. Journ.*, 1893, 698), who found that when solid calcium carbonate is left in contact with a solution of magnesium sulphate, or solid magnesium carbonate with a solution of calcium sulphate, magnesium sulphate is always eventually found in solution, and calcium carbonate is deposited.

TABLE X.

Lancashire Boiler, working pressure, 70 to 80 lbs.

	Feed Water.	Water in Boiler after working 82 hours.	Water in Boiler after working 468 hours.	The same Water evaporated at about 6 oz. per square inch.
Nitric acid (N_2O_5) ...	1·315	3·587	6·48	16·29
Chlorine (Cl)	1·70	15·75	37·0	37·00
Carbonic acid (CO_2) ...	8·68	4·49	6·07	1·99
Sulphuric acid (SO_3)	3·57	18·37	29·96	59·92
Silica (SiO_2)		5·10	8·35	2·88
Oxide of iron, etc. ...		0·4	0·32	0·76
Lime (CaO)	11·8	27·0	13·55	47·76
Magnesia (MgO) ...	0·81	0·43	0·18	3·37
Total solids	33·7	115·0	182·0	235·0

Table IX. contains the analytical results of two samples taken from a boiler, using New River water, at the expiration of the periods stated, together with those of the feed water. In order to get some idea of the difference of composition produced by evaporating at the ordinary pressure and at high pressure, a quantity of New River water was evaporated in the laboratory until it had the same *chlorine* figure as

the sample taken from the boiler after it had worked 468 hours. This was then analysed, and the results are placed side by side with those for the boiler sample of the same concentration.

The value of the figures is somewhat lessened by the fact that an organic boiler fluid was used in the boiler. Only 2½ gallons, however, was employed during the whole period, and it is probable that only the nitrates were in any way affected by it.

Of course, in the water evaporated in the laboratory, which was distilled from a 5-gallon tin can, which was replenished until the residue had the desired concentration, no boiler fluid was used.

Table X. gives the figures for the same samples after allowance has been made for evaporation in the usual way.

TABLE XI.

	Feed Water.	After 82 Hours.	After 468 Hours.	Evaporated at Ordinary Pressure.		Incrustation from Boiler. Parts per cent.
Nitric acid (N_2O_5) ...	1·32	0·39	0·30	0·75	moisture and organic matter	4·26
Chlorine (Cl)	1·70	1·70	1·70	1·70		
Carbonic acid (CO_2) ..	8·68	4·84	2·80	0·09		32·65
Sulphuric acid (SO_3) ...	3·57	1·98	1·38	2·75		4·42
Silica (SiO_2)	—	0·55	0·38	0·13		7·24
Oxide of iron, etc. ...	—	0·04	0·01	0·03		0·39
Lime (CaO)	11·8	2·91	0·62	2·19		44·84
Magnesia (MgO)	0·81	0·05	0·008	0·15		6·20
Total solids	33·7	12·41	8·36	10·8		—

The differences between the analytical results obtained from the samples of the same concentration evaporated at different pressure are much greater than would be expected. The N_2O_5 is between two and three times as great in the water evaporated at ordinary pressures, but this is no doubt partly accounted for by the presence of the boiler fluid; but, of course, it is quite possible, and indeed probable, that nitrates are more readily reduced by the metal of the boiler at high than at low temperatures. The tin can in which the *laboratory* sample distilled had lost most of the tin from its inner surface, so it is not surprising to find in Table X. that even here the nitric acid has been considerably reduced in amount.

The most surprising difference is found in the carbonic acid. The figures for it in this and in all the tables are really the *alkalinity expressed as* CO_2. (In the incrustations the CO_2 figures are the result of *direct* estimation.) There is actually less in the water evaporated at the ordinary temperature than in the sample taken from the boiler. As the estimations were done in duplicate, I am not disposed to attribute the discrepancy to error of analysis. A number of possible causes at once suggest themselves, but it would be unwise to speculate without further investigation.

The SO_3 is exactly double in the fourth column of what it is in the third. The difference here may fairly be put down to the high pressure and temperature combined affecting the solubility of the calcium sulphate.

The behaviour of the lime is almost as puzzling as that of the carbonic acid. In spite of the latter having almost entirely disappeared when the water was concen-

trated at ordinary pressure, three times as much lime remains in this sample as in that taken from the boiler. A glance at Table X., however, shows that the loss of lime when concentration takes place at ordinary pressure is mainly due to the deposition of calcium carbonate, the amount of sulphate of lime leaving the water being very small. At higher pressures it has already been seen that the latter salt becomes much less soluble.

Finally, the magnesia has diminished considerably less at the low than at the high pressure, which is quite in keeping with what happened in some of the other series of analyses.

The temptation to theorize and to draw conclusions even in the complicated phenomena under discussion is very great, but the series of analyses are not sufficiently extended and do not deal with waters of sufficiently varied types to justify it. They do, however, suggest many points of theoretical interest which require investigation, and may perhaps eventually pave the way to a clearer knowledge of the behaviour of salts in solution when heated under pressure.

I have to express my indebtedness to Mr. Charles E. Franck, my late assistant, and to Mr. F. W. Arnaud, for valuable assistance in carrying out the somewhat monotonous series of analyses.

Discussion.

Dr. Rideal said that an investigation of this kind ought to throw some light on the mineral constituents of natural water. The composition of natural water must depend upon the temperature and pressure at which the saline constituents came into contact with the water.

Mr. Jenkins desired especially to thank the author for the figures which the paper contained, which would be of great interest to many chemists. He would like to hear whether the author had any experience of an alkaline water, such as that referred to in Table V., showing, after evaporation, any evidence of caustic alkali. Dr. Paul had some considerable time ago recorded results indicative of the presence of caustic soda, as well as of carbonate of soda, in boiler water. It was within his own knowledge that water softened with a small quantity of soda (introduced as caustic soda, but fully carbonated before the water was put into the boiler) contained, after concentration in the boiler, small quantities of caustic alkali, as shown by titration with phenolphthalein and methyl orange. He had himself met with a case of salt scale from a land boiler which had to use water containing a considerable quantity of common salt. This scale contained about 80 per cent. of sodium chloride, most of the remainder being magnesium sulphate.

Mr. Allen said that a good deal of the effect of magnesium chloride was mitigated in the presence of an excess of sodium chloride, whereby it might be converted into a fairly stable double chloride of magnesium and sodium which did not decompose on evaporation. A similar reaction was to be observed in the case of magnesium chloride and ammonium chloride, a double chloride of magnesium and ammonium being formed which would stand evaporation. In fact, this was the explanation of the fact that sea water could be used in a boiler without serious corrosion taking place. Any calcium sulphate present in a boiler water (at any rate from a

high-pressure boiler) must be looked upon as absolutely insoluble, it being a well-established fact that calcium sulphate was insoluble in water under a pressure of, say, two additional atmospheres. Having regard to the conditions under which water could be softened by means of sodium carbonate, mixed perhaps with lime, he was driven to the conclusion that calcium carbonate was not insoluble in water under all conditions, as was supposed to be practically the case, and that it might often exist permanently in solution to a considerable extent, perhaps in a colloid condition, in which it was not readily precipitated by boiling. In instances within his recent knowledge, at least two or three times as much sodium carbonate had been added to water as was theoretically sufficient to precipitate all the calcium carbonate, and yet the water contained calcium carbonate representing a hardness of 7° or 8°, not capable of precipitation by an excess of sodium carbonate.

Mr. W. T. BURGESS inquired what was the source of the alkaline water referred to in Table V.

Mr. BEVAN said that he would like to hear how the carbonic acid had been estimated.

Mr. CHAPMAN said it was interesting to note how completely the magnesia seemed to have disappeared in two of the cases referred to. He would like to hear whether the author was of opinion that the whole of the magnesia had become converted by double decomposition into magnesium hydroxide.

The PRESIDENT (Mr. Fisher) said that he also had noted the alkaline water referred to in Table V., and had a certain amount of curiosity to know from what geological formation it came. It was a common experience to find alkaline water whenever a bed of clay was pierced at some distance from the outcrop of the porous beds. It seemed as though the magnesia disappeared as the carbonic acid disappeared from the water. Where the carbonic acid remained, as was indicated in Table V., it was bound by the potash and soda, and so the magnesia had not any control over it. It was interesting to notice how well the chlorine served as an index of concentration in connection with questions as to the formation of scale. The concentration which took place in the chlorine, where it was originally present in small quantities, afforded a valuable index of the ratio of feed water to evaporated water; but where the chlorine was higher originally than in most of the cases now referred to, there might not be quite so much certainty in applying that comparison.

Mr. CRIBB, in reply, said he saw no reason for supposing that caustic soda might not be present in boiler water as the result of some double decomposition, though he had not looked for it in the course of the present investigation. With regard to the question of the insolubility of calcium sulphate, all his analyses showed that, at any pressure at which he had experimented, a considerable quantity of calcium sulphate remained in solution. Had calcium sulphate been quite insoluble, all the lime should have disappeared, seeing that there was always more sulphuric acid present than was required by the lime; but in no case had this happened. The water referred to in Table V. came from the chalk under the London clay. The figures given under the heading "Carbonic Acid" were really the alkalinities of the waters expressed as CO_2, and were obtained by adding excess of standard acid and titrating back with alkali, using methyl orange as indicator. The end reaction was always considerably

obscured in the case of the very concentrated waters. He therefore could not claim any very great accuracy for the carbonic acid determinations in these cases. He was of opinion that the magnesia in the scale referred to in Table II. was in the form of oxide, and not of hydroxide, although in all the published analyses of scales he had seen the magnesia was put down as hydrate. It was pretty generally accepted that calcium sulphate was deposited as a hydrated sulphate, and afterwards underwent conversion into anhydrous sulphate, and in view of the high temperature to which one side of a thick incrustation would be exposed there was no reason why magnesia should not also become dehydrated.

Mr. JENKINS said that he had often found considerable quantities of calcium sulphate present in blow-off waters; and his experience was also that, in the case of such waters, the magnesia had nearly entirely disappeared. If one considered the magnesia as present in the form of carbonate rather than of chloride, the method of its precipitation became clear.

Mr. ARCHBUTT, who was unable to be present at the reading of the paper, has forwarded the following remarks:

One of the most interesting facts brought out by the paper was the very complete manner in which magnesia becomes precipitated in the boiler. This he was able to confirm from some analyses of his own. It seems probable that this is brought about by the calcium carbonate reacting with the magnesium salts, as shown by the following equation: $CaCO_3 + MgSO_4 + H_2O = CaSO_4 + Mg(OH)_2 + CO_2$; for Bohlig has shown that when calcium carbonate is boiled with a solution of magnesium sulphate, CO_2 is evolved, calcium sulphate is found in solution, and basic magnesium carbonate is precipitated. If the temperature be high enough, the precipitate consists of magnesium hydroxide (*Zeits. Anal. Chem.*, 1879, 195). The boiler scale in Table III. consists mainly of $CaSO_4$ and MgO; and as the original water contained a fair amount of carbonate, the whole of the precipitated calcium carbonate appears to have been used up in decomposing magnesium salts. It would be interesting to know what would happen in the case of a water entirely free from carbonates.

Another important fact confirmed by Mr. Cribb's experiments is that calcium sulphate does not, as commonly stated, become insoluble in water at 302° F., corresponding to a boiler pressure of 55 pounds per square inch. As Tilden and Shenstone's experiments related only to solutions of calcium sulphate in pure water and in water containing chlorides, it is satisfactory to find their results practically confirmed under conditions of actual boiler practice. It is a pity that in the interesting experiments contained in Table VIII. the heating at each temperature could not have been maintained for a longer period, but the results, confirmed by other analyses in the paper, seem quite to dispose of the contention that by merely heating hard water to 350° F. in closed heaters, without chemicals, the hardness can be reduced uniformly to 5 or 6 degrees, as has been stated. This statement can only be true of waters the hardness of which is mainly temporary. Nevertheless, it seems that after the removal of the temporary hardness a water containing a moderate amount of calcium sulphate might be prevented from forming scale by the judicious use of the blow-off cock and sufficiently frequent washing out of the boiler, so as to prevent the calcium sulphate from reaching the saturation point. Practical experience in many places seems to confirm this supposition.

The author's experiments and observations on the behaviour of magnesium chloride solutions when evaporated and distilled afford valuable information, and his promised further experiments on the behaviour of such solutions when distilled under pressure will be awaited with interest, as beyond the fact that waters containing much chloride and magnesia are found to be corrosive to boilers our knowledge is not as definite as it might be.

ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

FOODS AND DRUGS ANALYSIS.

Determination of Chloral Alcoholate in Chloral Hydrate. F. Schmidinger. (*Monatsh. Chem.*, 1900, xxi., 36.)—Meyer and Haffter (*Ber.*, vi., 600) have recommended a method which is quoted in the Commentary to the Austrian Pharmacopœia, depending on a titration of the chloral hydrate with normal sodium hydroxide. According to the equation

$$CCl_3CH(OH)_2 + NaOH = CHCl_3 + HCO_2Na + H_2O$$

100 parts of the pure hydrate should react with 24·17 parts of soda, whereas 100 parts of chloral alcoholate only require 20·67 parts of alkali. The process is useful to ascertain whether a particular sample is pure; but it can only indicate the presence of alcoholate provided other impurities not capable of reacting with sodium hydroxide are absent. The author finds that Zeisel's methoxyl method (*Monatsh. Chem.*, 1885, vi., 992) is better adapted for the examination of chloral hydrate, and that it gives satisfactory results. About 1 or 2 grammes should be operated upon.

This process is carried out by boiling the substance with 10 c.c. of hydriodic acid of specific gravity 1·68 in a current of carbon dioxide, the flask being attached to an inverted condenser which is supplied with water at 40° or 50° C. The vapours pass through bulbs charged with water and amorphous phosphorus maintained at 50° or 60° C., and are then absorbed in two flasks filled with an alcoholic solution of silver nitrate. After about two hours the whole of the iodine resulting from the methylic iodide which is produced by the action of the hydriodic acid upon the methoxyl compound is converted into silver iodide; the alcoholic solution is diluted with water, and the precipitate is weighed as usual. F. H. L.

Detection of Antifebrin [Acetanilide], Phenacetin, Exalgin, and *P*-amidophenol in Antipyrine or Quinine. P. N. Raikow and P. Schtarbanow. (*Oesterr. Chem. Zeit.*, 1900, iii., 125.)—Two processes are recommended: Distillation with phosphoric acid for the detection of the acid radicle and observation of certain changes in colour, and distillation with potassium hydroxide for the detection of the basic constituent. One gramme of the substance is cautiously warmed with 3 c.c. of

phosphoric acid, specific gravity 1·7, to a temperature short of that which decomposes the aniline phosphate, when antifebrin, phenacetin, and exalgin yield distillates of acetic acid. Antipyrine, antifebrin, and exalgin become more or less yellow or brownish yellow; phenacetin gives very characteristic colours: at first the mixture is pink, then wine-red, red-violet, violet, bluish green, and finally dirty green. If the liquid is cooled at the red-violet stage, it retains its colour unaltered for a long time, and this colour is not affected by the presence of antipyrine or antifebrin. The violet tint appears immediately antifebrin containing only 2 per cent. of phenacetin is boiled with phosphoric acid.

To detect the base, a few decigrammes of the sample are heated with 2 to 4 c.c. of strong aqueous potash, allowing the distillate to drop on to a little chloride of lime solution in a test-tube, which is changed (if necessary) after each single drop has fallen. Antifebrin gives the violet colour characteristic of aniline with its first or second drop, and the tint steadily becomes stronger. With phenacetin, the first drops remain colourless, subsequent ones yielding a brick red, due to the phenetidine, while on further heating the chloride of lime becomes turbid, and an amorphous red substance collects on the surface of the liquid, which finally turns yellow and clear again. Mixtures of phenacetin with 2 per cent. of antifebrin may be recognised by the different boiling-points of their basic constituent, changing the receivers so as to collect the pure aniline in one, and the phenetidine in the second, third, or fourth. When pure antipyrine is boiled with caustic potash, the chloride of lime remains colourless; but afterwards a substance distills which renders the solution milky, eventually forms white scales, and, combining with aniline and phenetidine, may prevent them yielding their characteristic colour reactions. This substance, however, which is neither antipyrine itself nor a chlorine derivative, is not produced till after the aniline from a mixture of antipyrine and antifebrin has passed over, so that 2 per cent. of the latter is easily to be found. Mixtures of antipyrine with phenacetin do not exhibit the proper red colour unless the proportion of the phenacetin is relatively high; the chloride of lime becomes yellowish green, then yellowish gray. A material composed of 0·1 gramme of antipyrine, 0·01 gramme of antifebrin, and 0·03 gramme of phenacetin, yielded the violet aniline colour with its first drop of distillate, which was changed to an orange by the next drops, so that both the latter bodies could be identified in the antipyrine. Distilled with potash, exalgin yields oily drops of methylaniline floating on the chloride of lime solution; these quickly turn green, and a dirty-brown precipitate appears. The test succeeds in the presence of antipyrine and antifebrin.

Quinine gives rise to no volatile compound on distillation with potassium hydroxide which is capable of affecting the solution of bleaching powder. Before testing with phosphoric acid the free base must be liberated from its salts; then the alkaloid yields no acid vapours, while the liquid in the retort first becomes yellow, then shows an intense "yellow-bluish-green" fluorescence.

Traces (0·0001 gramme) of *p*-amidophenol in these febrifuges are indicated by a deep violet colour when the sample is shaken with the phosphoric acid in the cold; the solutions ought to be colourless. F. H. L.

ORGANIC ANALYSIS.

Sugar Estimation in Zeiss's Immersion Refractometer. J. A. Grober (*Centralbl. inn. Med.*, 1900, xxi., 201 ; through *Chem. Zeit. Rep.*, 1900, 73.)—In the original article this apparatus is fully described, and its sphere of usefulness outlined. By observing saccharine urine before and after fermentation, the difference in reading divided by 2·9 gives directly the percentage of sugar; and by examining albuminous urine before and after precipitation with acetic acid, the difference divided by 0·3 gives the albumin in parts per 1,000. F. H. L.

The Solubility of Essential Oils in Sodium Salicylate. M. Duyk. (*Bull. de l'Acad. belge royale ;* through *Journ. Pharm. Chim.*, 1899, x., 500-504 ; *cf.* ANALYST, this vol., 72.)

Oil of Peppermint.—This yields to dilute salicylate solution (4 : 1·5) 78 per cent. of soluble products containing menthone and nearly the whole of the menthol. The insoluble portion consists principally of hydrocarbons (? menthene), and other compounds but little known.

The menthol contained in the soluble and insoluble portions may be estimated in the following manner : Several grammes of the substance are boiled for two hours under a reflux condenser with 10 c.c. of acetic anhydride and 2 grammes of sodium acetate, and the acetyl value calculated from the difference between the amount of sodium hydroxide required by the acetic anhydride used and that required after acetylation.

The amount of menthol is obtained by the formula

$$x = \frac{60 \times A}{156},$$

in which A represents the amount of acetyl which has entered into combination, and 60 and 156 the respective molecular equivalents of acetic acid and menthol.

Oil of Black Mustard.—This consists of allyl sulphocyanide, and is insoluble in the salicylate solution.

Oil of Bitter Orange.—The aldehydic compounds (citral, etc.), which are present in very small quantities, dissolve in the salicylate solution. The limonene remains undissolved.

Oil of Rosemary.—An oil of doubtful purity yielded to the salicylate solution 16 per cent. of substances, with a strong odour of camphor. The insoluble portion had a higher rotatory power than the original oil.

Rose Oil.—This is completely soluble in dilute salicylate solution (4 : 1). When adulterated with paraffin compounds it is only partially soluble.

Santal Oil (Indian) contains only traces of compounds soluble in salicylate solutions.

Oil of Turpentine.—When pure, this is insoluble in the salicylate solution; but when oxidized, the aldehydic compounds which have been produced are soluble.

The test is valuable in detecting the presence of turpentine in soluble essences.

Oil of Thyme.—The whole of the thymol and carvacrol are extracted by the salicylate solution, and when separated form a colourless syrupy liquid. The hydrocarbons are left undissolved. C. A. M.

Terpeneless Essential Oils of Lemon and Orange. N. Wender and G. Gregor. (*Chem. Zeit.*, 1900, xxiv., 210.)—The authors have examined samples of these oils which were described as being "terpeneless," obtained from three well-known firms, and they record their results herewith. The opticity was determined in a 100-millimetre tube at 20° C., using sodium light passed through a chromate solution; and owing to the high rotatory power of the oils themselves, they were tested in the form of alcoholic solutions, preliminary experiments having shown that no appreciable errors in the readings are introduced by the dilution. As the terpenes of the essential oils are less soluble in water than the oxidized constituents, experiments were also tried to discover a relationship between the water-solubility of the "terpeneless" oils and their rotatory power, these being carried out by shaking a 10 per cent. alcoholic solution in a tall, graduated cylinder at 15° C. with increasing quantities of water, till a homogeneous, faintly opalescent liquid was produced. The last column in the table indicates the volume of water in c.c. required to dissolve 1 c.c. of the 10 per cent. alcoholic solution.

	Mark.	Strength of Solution, Per Cent.	Opticity of Solution.	Opticity of Pure Oil.	Solubility.
Lemon Oil ...	H. H.	25·61	−1·90°	−7·41°	400
,, ,, ...	Sch. and Co.	25·09	−0·25°	−0·99°	500
,, ,, ...	O. W. and Co.	23·83	+1·75°	+7·34°	1,000
Orange Oil ...	H. H.	25·87	+3·40°	+13·10°	500
,, ,, ...	Sch. and Co.	25·97	+16·60°	+63·91°	1,000
,, ,, ..	O. W. and Co.	20·85	+14·50°	+69·70°	1,200

Especially in the case of terpeneless orange oil, the solubility in water falls as the rotatory power rises; but it is clear that these oils are still of very uncertain composition, even when procured directly from reputable firms. F. H. L.

Estimation of Chromium in Chrome-tanned Leather. P. von Schroeder. (*Deutsche Gerber Zeit.*, 1899, December; through *Zeits. angew. Chem.*, 1900, 142.)—About 3 grammes of the leather are thoroughly ignited for two or three hours in a porcelain crucible. The residue is moistened with 60 per cent. nitric acid (specific gravity, 1·37), and solid potassium chlorate is added. The crucible is heated on the water-bath with fresh additions of acid and chlorate until the solution becomes red, when it is evaporated to dryness two or three times with strong hydrochloric acid. The solid matter is then washed into a porcelain basin by means of water, a few drops of dilute sulphuric acid are introduced, and sodium peroxide is cautiously dropped in until the green liquid changes to pure yellow. The hydrogen peroxide (which would reduce the chromic acid when the solution was acid, but which is inert in presence of alkali) is next removed, either by simple evaporation or by means of a little alcohol; the whole is mixed with 25 c.c. of 10 per cent. solution of potassium iodide and 25 c.c. of 1 : 5 sulphuric acid, and the iodine liberated is titrated with decinormal thiosulphate. Seven samples of chrome-tanned leather gave 0·82, 1·68, 1·43, 3·4, 2·9, 3·22, and 2·25 per cent. of metallic chromium respectively; but it should be noted that there appears to be no connection between the proportion of chromium and the quality of the leather. F. H. L.

INORGANIC ANALYSIS.

The Gas-Volumetric Estimation of Hydrogen. A. Colson. (*Ac. des Sciences*, 1900, cxxx., 330; through *Journ. Pharm. Chim.*, 1900, xi., 335.)—This method is based upon the fact that hydrogen is absorbed by silver hydroxide. The reaction takes place slowly in the cold, but at 100° C. is rapid, and furnishes a means of separating hydrogen from a saturated hydrocarbon or from oxygen.

The mixed gases are introduced into a eudiometer, provided near the top with a horizontal side-tubulure, in which has previously been placed 1 to 2 grammes of silver hydroxide. On heating the side-tubulure the mercury rises in the eudiometer, and the absorption is complete in two and a half to three hours. In calculating the true volume of the residual gas, allowance must be made for the tension of the water-vapour formed during the reaction.

Hydrogen is completely absorbed, even at a very low pressure, whilst, as the author shows in his test estimations, no absorption of ethane, methane or oxygen takes place. C. A. M.

The Estimation and Separation of Copper by means of Hydrazine Sulphate or Hydrazine Hydrochloride. P. Jannasch and K. Biedermann. (*Berichte*, 1900, xxxiii., 631-636.)

Estimation of Copper.—The hot solution of the copper salt is poured, with constant stirring, into a porcelain basin containing a solution of 5 grammes of pure sodium hydroxide in 50 c.c. of water. The resulting precipitate is reduced to cuprous hydroxide by adding 1 to 2 c.c. of a boiling 3 per cent. solution of hydrazine sulphate, and slowly heating the basin on the water-bath while its contents are continually stirred. On the further addition of 3 c.c. of the hydrazine sulphate solution the copper is reduced to the metallic state. After cooling, or diluting the liquid with boiled water, the precipitate is collected on a filter, washed with hot water, dried at 90° C., the paper ignited apart from the copper, and the latter oxidized over a Bunsen flame in a current of oxygen and weighed.

The results thus obtained in test experiments with copper sulphate were satisfactory.

Separation of Copper from Zinc.—In the authors' experiments the solution of the mixed sulphates was introduced drop by drop into a basin containing 50 c.c. of 10 per cent. sodium hydroxide solution. Five c.c. of a 3 per cent. solution of hydrazine sulphate were then added to the cold liquid containing a large excess of sodium hydroxide, and the whole slowly heated on an asbestos board. After about five minutes there was a complete precipitation of the copper, which was collected and oxidized as described above.

The filtrate containing the zinc was treated with hydrochloric acid, until the precipitate, which formed after some time, again dissolved, and the liquid was distinctly acid. The zinc was then precipitated as carbonate, which was ignited and weighed in the usual manner. Sometimes the zinc oxide obtained was not completely soluble in dilute acetic acid owing to the presence of traces of silica.

In this way the following results were obtained :

In Solutions used Per Cent.		Found Per Cent.	
Copper.	Zinc.	Copper.	Zinc.
16·77	7·74	16·73	7·68
15·69	8·68	15·61	8·68

Separation of Copper from Arsenic.—A solution of copper sulphate and arsenic acid was introduced drop by drop into 40 to 50 c.c. of 10 per cent. sodium hydroxide solution, the liquid warmed after the introduction of 4 to 5 c.c. of hydrazine sulphate solution, and the precipitated copper estimated as before.

The solution containing the arsenic was acidified with hydrochloric acid, a little nitric acid being added with it towards the end of the neutralization, and the whole evaporated to about 80 c.c., care being taken to have some nitric acid continually present. The clear liquid when cold was treated with ammonium hydroxide in excess, and the arsenic precipitated with freshly-prepared magnesia mixture, and estimated in the usual way. Results :

Taken Per Cent.		Found Per Cent.	
Copper.	Arsenic.	Copper.	Arsenic.
17·53	23·41	17·46	23·37
13·68	35·29	13·56	35·24

Separation of Copper from Tin.—Mixtures of pure copper and tin were dissolved in *aqua regia*, the solutions diluted with an equal volume of water, and added drop by drop to a hot solution of sodium hydroxide containing fifteen times more alkali than the weight of the dissolved metals. After the addition of 2 to 3 grammes of hydrazine hydrochloride the liquid was heated, and the heating continued for some time, in order to dissolve as sodium stannate any tin carried down with the copper. In the absence of sulphates any tin readily dissolved. The copper was collected on a double filter, washed with boiling water, and if necessary with dilute sodium hydroxide solution and again with water, dried, oxidized and weighed.

The filtrate was rendered slightly acid with strong hydrochloric acid, and the tin precipitated with ammonium hydroxide. The precipitate was dissolved by adding ammonium sulphide, and the liquid heated for a time on the water-bath and then slightly acidified with hydrochloric acid. After being heated for about an hour on the water-bath, the resulting precipitate of tin sulphide subsided and could be easily filtered. It was washed with warm hydrogen sulphide solution, ignited in a current of oxygen and weighed. The following results were obtained :

Taken Per Cent.		Found Per Cent.	
Copper.	Tin.	Copper.	Tin.
25·81	74·18	25·72	74·06
31·28	68·61	31·28	68·48

C. A. M.

The Analysis of Molybdenum Alloys. H. Borntträger. (*Zeit. anal. Chem.*, 1900, xxxix., 91.)—As a supplement to his method of analysing molybdenum glance

(ANALYST, xxiii., 332), the author describes the following process for determining molybdenum in its alloys:

About 1 gramme of the sample is dissolved in 50 c.c. of *aqua regia* on the water-bath, the solution evaporated in a porcelain basin, and the nitric acid expelled by means of hydrochloric acid.

The residue is taken up with 50 per cent. alcohol, and the liquid filtered. The residue left on the filter will now contain the bulk of the molybdic acid.

The filtrate is evaporated to dryness, and the residue once more taken up with alcohol, after which treatment the remainder of the molybdic acid will be left as a residue.

The molybdic acid in the two residues can then be weighed directly, whilst the metal in the solution can be estimated in the usual way as oxide.

From an alloy known to contain 66 per cent. of molybdenum and 44 per cent. of iron, the author obtained by this method 65·6 per cent. of molybdenum. C. A. M.

The Detection of Boric Acid in the Form of Borates. H. Borntträger. (*Zeit. anal. Chem.*, 1900, xxxix., 92.)—When borates are heated with hydrochloric, nitric, or sulphuric acid, a Bunsen flame is not coloured green. On the other hand, when heated with hydrofluoric acid alone, or with ammonium nitrate and ammonium chloride, or with sulphuric acid and hydrochloric acid, or sulphuric acid and nitric acid, or hydrochloric acid and nitric acid, borates impart a bright green colour to the flame; and this coloration is more intense, and appears sooner, than that obtained on heating the salts with alcohol and sulphuric acid. C. A. M.

A New Method of estimating Bromides in the Presence of Chlorides and Iodides. J. v. Weszelszky. (*Zeit. anal. Chem.*, 1900, xxxix., 81-91.)—Bromine can be determined in the presence of chlorine by taking advantage of the fact that in an alkaline solution it is oxidized by chlorine to bromic acid, as in the equations:

$$Br + 6KOH + 5Cl = KBrO_3 + 5KCl + 3H_2O, \text{ and}$$
$$KBr + 6KOH + 3Cl_2 = KBrO_3 + 6KCl + 3H_2O.$$

The excess of chlorine is converted into chloride and chlorate; but as the solution of the latter salt is too weak to act upon iodides in the cold, the amount of bromate formed may be determined by adding a solution of potassium iodide, acidifying, and titrating the liberated iodine.

For the separation of bromine and chlorine from iodine, the author makes use of Winkler's method, which is based upon the fact that chlorine oxidizes iodine or iodides in acid solution to iodic acid, whilst the bromine is only liberated. Thus, on acidifying a solution of the mixed halides, adding an excess of chlorine water, and boiling the liquid, the resulting iodic acid remains behind, while the bromine and excess of chlorine distil over. On adding potassium iodide to the cooled liquid in the flask, and titrating with $\frac{N}{10}$ thiosulphate, a sixth part of the iodine found corresponds to the iodine originally present.

In applying these reactions, if the solution contains no iodine, about 1 gramme

of potassium carbonate is introduced, together with a sufficient quantity of chlorine water, and the liquid cautiously evaporated to dryness over a naked flame. The residue, when cold, is dissolved in 100 to 150 c.c. of water, potassium iodide added, and the liquid acidified and titrated with $\frac{N}{10}$ thiosulphate. The number of c.c. used, multiplied by 0·001333, gives the quantity of bromine.

When iodine is present, the liquid is placed in a distilling flask, similar to that used by Bunsen and Fresenius in the analysis of manganese peroxide (Fresenius, "Quantit. Analyse," 6th edit., i. 382). This is connected with an absorption vessel, containing 0·5 to 1·0 gramme of potassium hydroxide dissolved in water. After the addition of a sufficient quantity of chlorine water, the flask is warmed, and when the greater part of the chlorine and bromine has passed over, a current of carbon dioxide is passed through the apparatus until the end of the distillation.

The bromine in the distillate and the iodine in the flask are determined by titration with thiosulphate, as described above.

When the solution of the halogen contains iron, the latter should be separated by precipitation with potassium carbonate; arsenic and antimony when present must also be removed.

The author gives tables of the results of test analyses of experimental mixtures of bromides, iodides, and chlorides, from which it appears that the method is extremely accurate, even when only minute quantities of the halogens are present.

C. A. M.

The Determination of Clay in Soils. F. Poquillon. (*Bull. Soc. Chim.*, 1900, xxiii., 115, 116.)—The author recommends the following method as being much more rapid than the usual process, requiring at the outside only two or three days.

Ten grammes of the soil are triturated with about 25 c.c. of water added drop by drop, and 100 to 120 c.c. of a solution of ammonium chloride (1 gramme per litre) added to the mixture. The mass is stirred with a glass rod, and after standing for five minutes the supernatant liquid is transferred to a litre flask. The residue is again treated with 100 to 125 c.c. of the ammonium chloride solution, and the liquid again decanted after five minutes, this process being repeated until the washings are clear. About six or eight washings are required in the case of heavy clay soils. The residue is then treated with dilute hydrochloric acid, washed with water, dried, and weighed, the weight giving the amount of total sand.

The liquid in the flask is treated with a few drops of hydrochloric acid to dissolve carbonates and to coagulate the clay, after which it is left until the supernatant liquid is clear. This usually takes two or three hours. The deposited clay is then collected on a weighed filter, washed with water, dried, and weighed.

The following results were obtained by this method, and by the ordinary method, which takes from eight to ten days:

Clay in the Soil.—Parts per Thousand.

Old method	... 206·0	60·2	198·0	218·0	122·0	129·3
New method	... 205·3	60·5	198·0	217·1	121·9	129·5

C. A. M.

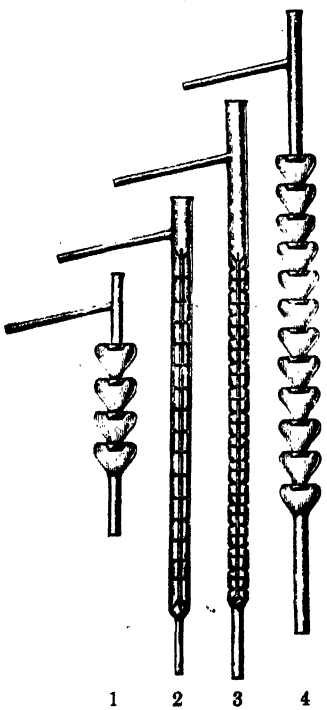
A New Indicator: Alizarin Green B. J. Formánek. (*Zeit. anal. Chem.*, 1900, xxxix., 99-103.)—Alizarin Green B, which is manufactured by Dahl and Co., Barmen, is obtained by the action of β-naphtho-quinone-sulphonic acid on (2)-amido-(1)-naphthol-(4)-sulphonic acid.

It is a greenish-black powder, which dissolves fairly readily in water, forming a dirty-green solution. It is somewhat less soluble in alcohol, the colour of the solution being flesh-red. On adding a dilute acid to the alcoholic solution the colour becomes carmine red, whilst alkalies change it to a pure green. Like litmus, it is sensitive to carbonic acid.

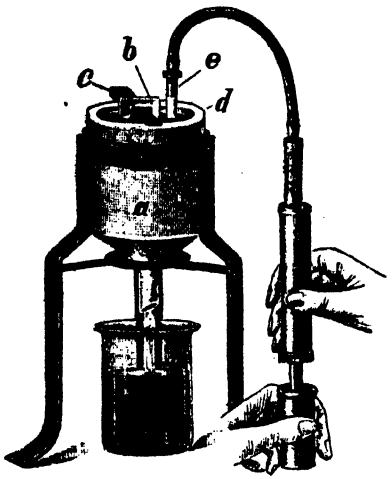
The changes in the colour reactions are exceedingly sharp, and are as perceptible with an artificial light as in daylight. C. A. M.

APPARATUS.

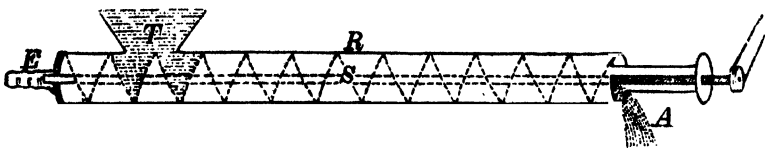
Some Improved Still-heads. S. Young. (*Journ. Chem. Soc.*, 1899, lxxv., 679.)—In this article the author draws attention to the various defects of the popular dephlegmators, and shows that the Glinsky tube (glass balls) is inferior even to the Le Bel Henninger (platinum gauze and lateral reflux tubes). He then describes two new forms of apparatus, which are illustrated herewith. Figs. 1 and 4 represent the "pear" head of different sizes. In this the liquid condensed in any particular bulb drops vertically through the lower ones without travelling over the surface; and thus the quantity of condensed fluid is diminished. Figs. 2 and 3 show the "rod and disc" heads. The discs are flanges of glass constructed on a glass rod by "upsetting" it at the necessary places. In Fig. 2 the main tube is parallel, and the rods-and-discs can be withdrawn; in Fig. 3 the tube is provided with constrictions, which increase the efficiency of the device. To obtain fractionation equivalent to that yielded by a three-bulb Le Bel Henninger tube, the rod-and-disc apparatus, or preferably the "pear" tube is advocated. They are simpler, better suited for the treatment of small volumes of liquid, and at the end of the process return almost all the residue to the still [while, as the makers point out, the "pear" head enables the retort to be recharged without disarranging the whole apparatus, and without danger of breaking the tube]. For an efficiency equal to that of a Young and Thomas dephlegmator of 12 or 18 columns, however, the new heads are too long; and therefore either the former device, modified according to the illustration on p. 700 (*loc. cit.*), should be retained, or preferably an "evaporator" still-head (p. 696) should be adopted. The improved heads are made by J. J. Griffin and Sons. F. H. L.



A Laboratory Filter Press. **W. v. Loeber.** (*Chem. Zeit.*, 1900, xxiv., 193). —This apparatus is specially suitable for the filtration of hot or volatile liquids where the ordinary suction-pump exhibits certain disadvantages. *a* is a cylindrical porcelain vessel carrying a perforated false bottom just above its conical base. At the top it is fitted with a flange, the internal edge of which is elliptical in shape; and the cover, also elliptical, is of larger area than the aperture. The cover has an open handle (*b*), through which a wooden wedge (*c*) is driven, and this arrangement pulls it upwards against the inner surface of the flange, making a tight joint through the agency of a rubber washer. [This part of the device is precisely analogous to the "manhole" of a steam boiler.] The tube *e* is closed at its internal end, but has a lateral opening; a piece of rubber tube is slipped over it to form a non-return valve. Pressure is applied by means of a pneumatic tyre inflator. In using the apparatus a moistened filter-paper is laid on the perforated bottom; and if the liquid is such that the paper does not lie flat, the two halves of a split porcelain ring are placed on it to hold it down. The cover is then brought within the flange at right angles to its proper position, held lightly with the wedge, and the liquid is run in through a funnel; the cover is next rotated to its correct position, tightened up, and the pressure applied. If the precipitate is one of those that fissure before they are fairly dry, thus permitting escape of air, when the bulk of the filtrate has run through, the apparatus is opened, another paper is laid on the top of the precipitate, the cover is wrapped in a thin sheet of rubber, *e.g.*, a piece of a toy air-balloon, and the joint is made as before. On pumping up, the rubber expands till it fills the space of the filter, pressing the top paper downwards on to the precipitate, and thus acting as a press. To release the pressure it is then necessary to puncture the rubber from beneath by means of a needle; but the hole in the sheet can be afterwards mended with tyre solution. A cyclist's pressure-gauge can be added if desired. The machine, made in two sizes, 250 c.c. and 2 litres, is on sale by the Actienges. für pharmaceut. Bedarfsmittel, Cassel, and has been protected as a "Gebrauchsmuster" in Germany. F. H. L.



A Laboratory Apparatus for igniting Large Quantities of Substances. **G. P. Drossbach.** (*Berichte*, 1900, xxxiii., 486.)—By means of the simple device shown



in the figure several kilogrammes of solid matter in the form of powder may be ignited in any gas required.

It consists of a tube (*R*), provided with a funnel (*T*), and a spiral conveyor (*S*), driven by a motor. The solid substance is introduced through *T* into the heated tube, and the gas at *E*, while the ignited product is discharged at *A*.

If the tube is 30 centimetres long and has a transverse section of 1 square c.c., and the spiral revolves once in a second, the substance will remain for a minute in the hot tube. C. A. M.

DIVISION OF SAMPLES UNDER THE SALE OF FOOD AND DRUGS ACT.

QUEEN'S BENCH DIVISION.

(Before Mr. JUSTICE DARLING and Mr. JUSTICE BUCKNILL.)

MASON *v.* COWDARY.

(*From the* PHARMACEUTICAL JOURNAL, *June* 9, 1900.)

THIS was a special case stated by the Justices of Bedford. It stated that, at a Petty Sessions held at Luton, January 8, 1900, an information was preferred by Geo. Mason, under Section 6 of the Sale of Foods and Drugs Act, 1875, against Ellen Cowdary, that she on November 16 did unlawfully sell to the prejudice of the appellant camphorated oil which was not of the nature, substance and quality demanded. The respondent kept a small shop in the village of Leagrave, Beds, and the appellant purchased from her six twopenny bottles of camphorated oil. The oil was exposed for sale in bottles which were not apparently prepared by respondent, but each of them bore a label with the name of a chemist in the neighbouring town of Luton. There was no evidence whether or not the bottles were identical in character or appearance, or whether or not the labels all bore the name of the same chemist, but the six bottles were all purchased at the same time. At the time of the purchase, the appellant intimated to the respondent his intention to have the oil analysed, and he divided the six bottles into three lots of two bottles, each of which he sealed up, handing one lot to the respondent, keeping one lot, and sending the other lot to be analysed. Appellant did not open any one of the bottles of oil, or mix or divide the contents; but the two handed to the respondent were in the same condition as when purchased. The appellant received from the Public Analyst a certificate of the analysis of the contents of the two bottles submitted to him, and the analysis was put in evidence. It showed that the camphorated oil in the bottles analysed contained only 17·5 per cent. of camphor, whereas proper camphorated oil should contain 20 per cent. of camphor. For the part of the appellant, it was contended that the requirements of Section 14 of the Sale of Food and Drugs Act had been complied with by him, inasmuch as he had divided the article into three parts, each of which was marked and sealed; but the justices were of opinion that the appellant had not complied with the requirements of Section 14, and, further, that they were not satisfied that the two bottles analysed by the analyst were identical in nature and substance with the other two sets of bottles in the hands of the seller and the appellant respectively. They accordingly dismissed the summons.

Mr. BONSEY said the appeal had been made because the appellant wished the point decided whether or not it was necessary to divide up each bottle in order to comply with Section 14.

Without calling on the other side, their lordships held this was a purchase of six separate articles, and therefore each required dividing and analysing. The appeal was therefore dismissed with costs.

APPOINTMENT.—Mr. Norman Leonard, B.Sc., F.I.C., has been appointed Public Analyst for the Borough of Northampton, *vice* Mr. Raymond Ross, resigned.

THE ANALYST.

AUGUST, 1900.

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ON AN IMPROVED ABSORPTION APPARATUS FOR USE IN THE ANALYSIS OF ESSENTIAL OILS.

By Alfred C. Chapman, F.I.C., and H. E. Burgess.

(*Read at the Meeting, April* 4, 1900.)

Absorption methods involving the removal of certain active constituents of essential oils by means of appropriate reagents, and the subsequent measurement of that portion which remains unabsorbed, are of very frequent application in laboratories where these oils are examined. As examples, the estimation of citral in lemon and lemongrass oils, cinnamic aldehyde in oil of cassia, and eugenol in oil of cloves, may be mentioned.

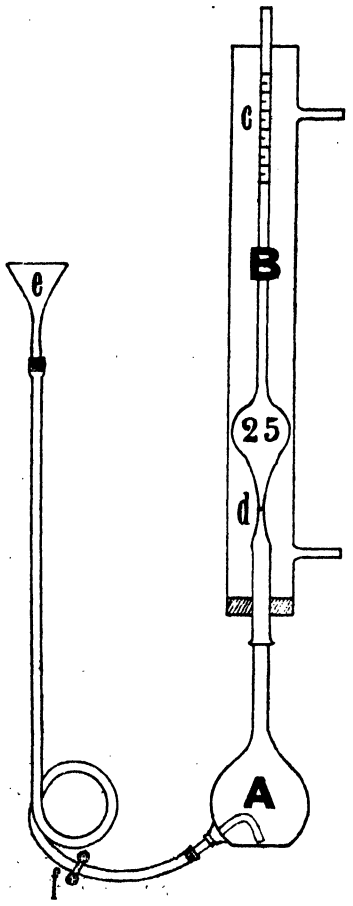
The instrument almost invariably used for such determinations is a simple glass flask capable of holding about 200 c.c. Its neck has such a capacity that it will hold 25 c.c. of the oil, and the space between the zero and the 25 c.c. mark is graduated in $\frac{1}{5}$ths, and sometimes $\frac{1}{10}$ths, of a c.c.

This piece of apparatus is known as a Hirschsohn flask, and there are many objections to be urged against its use. In the first place, it is practically impossible to treat the oil under examination with a second quantity of the reagent, or to wash it; it is, moreover, impossible, as a rule, to shake the mixture in the flask with sufficient vigour for fear of obtaining an emulsion, which will not readily separate, and any insoluble matter, such as is frequently formed on shaking the oil with the necessary reagents, accumulates at the junction of the two liquids, thus obscuring the meniscus and reducing the accuracy of the reading, an error which is further increased by the closeness of the graduations, due to the width of the neck.

It was to obviate to some extent these objections that we devised the piece of apparatus represented in the accompanying figure.

It consists essentially of two parts, the flask A of about 250 c.c. capacity, and

the measuring tube B. The latter is accurately ground to fit the flask A, and is arranged so that the volume intervening between the zero mark of the scale C and the mark *d* is exactly 25 c.c. The portion of the tube above the bulb has a considerably smaller diameter (about 5 millimetres) than that below, and is graduated downwards in 0·1 c.c., from 0 to, say, 5. The flask A has a tubulure at the bottom, through which passes a glass tube connected by indiarubber tubing to the funnel *e*. Communication between the two may be closed or established by means of the pinch-cock *f*.



In using the apparatus, 25 c.c. of the oil to be analysed is pipetted at a known temperature into the flask, and is there treated with a sufficient quantity of the necessary reagent, the pinch-cock *f* being closed. When the soluble portion has been dissolved, the aqueous layer may be run off by opening the pinch-cock, and hot or cold water for washing purposes may be run in through the funnel *e*, or a further quantity of the reagent may be introduced if it is thought that the absorption has not been quite complete.

The measuring tube is then fixed in position and the residual washed oil is transferred to it by running cold water through the funnel. The tube is next fitted with a cylindrical glass jacket, water is poured into, or circulated through, it, and the oil brought to the temperature at which it was originally measured out. Then, by raising or lowering the funnel, the meniscus is brought exactly to the lower mark, and the reading on the narrow portion of the tube taken, from which the percentage of the absorbed constituent can be at once determined.

It is obvious that several measuring tubes of different shapes and sizes may be fitted to the same flask, the character of the tube depending in each case on the precise purpose for which it is to be used.

The tube represented in the figure is suitable for the estimation of citral in oil of lemons, or for the examination of any other oil in which the soluble constituent does not occur to the extent of more than about 20 per cent.

This apparatus has now been in constant use for some months, and during that time many determinations have been made, working on artificially-prepared oils containing known percentages of the soluble constituents, with very satisfactory results.

Since our communication was made to the Society, we have seen in a paper by Soldaini and Berté a description of a piece of apparatus intended for the analysis

of lemon oil, which is very similar to ours in principle, but in which only 5 c.c. of the oil are used. So far as we are able to judge from the description, however, we are inclined for several reasons to give the preference to the apparatus described in this paper.

We may add that the complete apparatus may be obtained from Messrs. C. E. Müller and Co., 148, High Holborn, London, W.C.

Mr. CHAPMAN, in reply to a question put by Dr. Lewkowitsch, said that he had had experience of the direct method of determining eugenol, based on the conversion of the eugenol into benzoyl-eugenol, and had found that this method involved errors rather greater than those of estimation by difference.

ON THE COMPOSITION OF DANISH BUTTER.

BY HARALD FABER.

(*Read at the Meeting, April* 4, 1900.)

THE following few remarks on the composition of Danish butter were originally intended only for use in the discussion of a paper by Mr. Estcourt on "Butter from Various Countries Compared."

Very full information of the amount of water in Danish butter is obtained by the numerous analyses made in connection with the State Butter Shows in Copenhagen (Foreign Office Report, 1889, Miscellaneous, No. 151). Table I., showing the amount of water found by the analysis of about 12,000 samples during nine years, calls for very few remarks. The average is about 14 per cent., most samples having from 13 to 15 per cent., and very few less than 12 per cent., or more than 16 per cent.

TABLE I.

AMOUNT OF WATER IN DANISH BUTTER.

According to analyses of samples at the State Butter Shows.

Yearly Averages.

	Number of Samples.	Percentage of Water.
1890-94	4,458	14·32
1895	1,517	13·70
1896	1,992	13·68
1897	2,016	13·79
1898	2,110	13·93

Samples grouped according to Amount of Water.

Percentage of Water.	Number of Samples as Percentage of Total Number.	
	1897.	1898.
10-11	0·8	0·4
11-12	4·9	2·4
12-13	17·1	12·6
13-14	37·4	37·0
14-15	29·7	35·3
15-16	8·5	10·3
16-17	1·2	1·7
17-18	0·4	0·3

The Customs have for some years taken samples of butter on landing. Duplicates of the samples of butter from Denmark have been analysed by Professor Stein at Copenhagen. From his reports I have compiled the following table (Table II.), showing monthly averages for the years 1898 and 1899 of the Reichert-Wollny figure, the refraction and also the number of samples which responded to the Becchi test. The refraction is by Zeiss's refractometer at 45° C.

The chemical composition of the butter-fat as expressed, for instance, by the Reichert-Wollny figure varies with the season, the time since calving, and the treatment generally of the cows. In Denmark cows are generally housed from the middle of October to the middle of May, and the calving takes place in autumn and winter. Correspondingly, we find the Reichert-Wollny figure higher in the winter and spring than in summer, and lowest in the autumn, the maxima (32·6 and 32·5) for the two years occurring in March, the minima (24·3 and 25·6) in October. The effect of moving the cows from cold fields to warm byres in October, additional to the increased number of new-calved cows, finds its expression in a rapid rise in the Reichert-Wollny figure from October to November.

It has at last been recognised, also by the chemists of the Government Laboratory, that the feeding of cotton-cake imparts something to the butter which reacts to the Becchi test, and that, consequently, a cotton-oil reaction does not necessarily imply an addition of margarine. The comparison of the number of samples for the two years which gave the Becchi reaction is interesting. In 1898, which was remarkable for a luxurious growth of grass, the cows were grass-fed all through the summer, and from June to October inclusive not one sample exhibited the reaction, as no cotton-cake was used, while several samples reacted during the time the cows were stall-fed. The year 1899 was quite different; a cold and dry spring and a dry and hot summer affected the growth of grass so unfavourably that the cows had to have some additional food nearly all through the summer, and as cotton was the cheapest cake, and Russian sunflower cake (which is otherwise very much used) difficult to get, large quantities of cotton-cake were used, with the result that every fourth sample gave the Becchi reaction in summer-time, and six out of every seven responded to the test during the months of November and December.

TABLE II.

STATEMENT SHOWING VARIATION IN CHEMICAL COMPOSITION OF DANISH BUTTER.*

The samples analysed are duplicates of those taken by Her Majesty's Customs at port of importation.

1898.

Month.	Number of Samples.	Reichert-Wollny Figure.	Refraction. Zeiss at 45° C.	Becchi Test.	R.-W. Max. and Min.
January					
February ...	7	30·7	40·4	4	
March ...	16	30·2	40·0	10	Max. 32·6
April ...	None	—	—		
May	14	30·1	40·5		
June	3	30·2	40·7		
July	None	—	—		
August ...	7	29·1	41·8		
September ...	16	28·4	42·5		
October ...	6	26·4	42·7	,,	Min. 24·3
November ...	16	29·5	40·5	5	(Max. 31·7)
December ...	(2)	(30·0)	(39·4)	(1 ?)	

1899.

January ...	None	—	—	—	
February ...	8	30·7	40·0	7	
March ...	17	30·8	40·4	14	Max. 32·5
April	(2)	(28·3)	(41·5)	(None)	
May	22	29·3	40·7	14	
June	(2)	(30·5)	(40·6)	(1)	
July	None	—	—	—	
August ...	9	28·1	41·6	1	
September ...	13	26·9	41·8	3	Min. 25·6
October ...	3	27·9	41·2	2	(Max. 31·8)
November ...	18	30·8	39·9	15	
December ...	3	30·5	40·0	3	

PERCENTAGE OF NUMBER OF SAMPLES WHICH REACTED BY THE BECCHI TEST.

	1898.	1899.
January to May	62	71
June to October	0	26
November to December ...	33	86

* The figures included in brackets refer to samples which were so small that it was not deemed advisable to include them in the percentage table.

THE POLARIMETRIC ESTIMATION OF CAMPHOR IN CAMPHORATED OIL.

By Norman Leonard, B.Sc., F.I.C., and H. Metcalfe Smith, F.I.C.

In a previous note on the examination of liniment of camphor (*Analyst*, 1898, xxiii., 272) we described two methods for the estimation of camphor in camphorated oil, one of which, yielding only approximate results, depended on a determination of the specific gravity of the liquid, whilst in the second and more accurate method the camphor was determined from the loss of weight on heating. Further experiments have shown us that by means of the polariscope the assay of camphorated oil may be effected both accurately and expeditiously.

The rotatory power of camphor in its solutions in alcohol, benzene, and some other organic liquids has been studied by Landolt and others, but, so far as we are aware, no experiments have been made with olive or other oils as solvents. We have therefore examined a number of solutions prepared by digesting known weights of camphor and olive oil together at the ordinary temperature until complete solution had been effected. In the table below are given: (1) The weight of camphor contained in 100 parts of the solution; (2) the specific gravity of the liquid at 60° F., compared with water at the same temperature; (3) the angular rotation for sodium light observed in a 200-millimetre tube with a Schmidt and Haensch half-shadow instrument; (4) the amount by which the specific gravity is raised by each 1 per cent. of camphor; (5) the angular rotation per 200 millimetres for each 1 per cent. of camphor. The olive oil used in the preparation of the solutions had a specific gravity 0·91666, and a rotatory power +0·13° per 200 millimetres. The latter figure was deducted from the observed rotations of the solutions in calculating the numbers in the fifth column of the table. The last solution was a saturated one prepared by gently warming olive oil with excess of camphor, cooling to about 10° C., and filtering; the camphor in it being determined from the loss of weight sustained on heating at 110° to 120° C.:

1. Camphor per cent.	2. Specific Gravity at 60° F.	3. Rotation per 200 Millimetres.	4. Specific Gravity raised per 1 per cent.	5. Rotation per 200 Millimetres per 1 per cent.
5·32	0·91903	+ 5·26°	0·000446	0·964°
11·26	0·92173	+ 11·35°	0·000450	0·996°
20·66	0·92604	+ 20·74°	0·000454	0 998°
26·78	0·92911	+ 26·79°	0·000465	0··996°

It will be seen from these results that the rotation is increased by nearly 1° for each per cent. of camphor present, and that the observed rotation of a sample of camphorated oil in a 200-millimetre tube gives at once, without calculation, the percentage of camphor with sufficient accuracy for most purposes. The rotation appears not to be appreciably influenced by ordinary variations of temperature. According to Bishop (*Journal of the Society of Chemical Industry*, 1887, p. 750), olive oil has a rotatory power +0·13° per 200 millimetres, and several specimens examined by us have given values closely approximating to this, values too small to affect

seriously the optical estimation of camphor dissolved in such oils. In the case of some other oils which are occasionally substituted for olive oil in liniment of camphor the rotatory power is greater. Thus, we have found the rotation of two specimens of rape oil to be $-0{\cdot}16°$ and $-0{\cdot}3°$, and of sesame oil $+1{\cdot}6°$, per 200 millimetres, results which are in accord with those of Bishop, whilst two samples of fluorescent mineral oil gave rotations of $+0{\cdot}12°$ and $+0{\cdot}42°$. Experiments with rape oil and mineral oil have shown us, however, that the increased rotation caused by the solution of camphor in the oils is practically the same as with olive oil, so that if the nature of the oil used in the preparation of the sample is known, the process is still applicable. With regard to the identification of the oil used, we have found that the sample may be examined directly with the refractometer, the refractive index of the oil being apparently almost unaffected by the presence of dissolved camphor.

It is worthy of notice that the specific rotation of camphor in its solutions in olive oil is shown by our experiments to be about $+54°$, a higher result than is given by any of the solvents examined by Landolt, and approximating to the value ($+55{\cdot}4°$) calculated by him for the absolute specific rotation of camphor.

Since writing the above, our attention has been called to an account of some experiments by E. Dowzard (*British Food Journal*, 1900, p. 69), who finds, by comparing the rotatory power of a number of samples of camphorated oil with the amount of camphor "ascertained by the gravimetric process," that the angular rotation per 100 millimetres multiplied by the factor 1·962 gives the percentage of camphor in the sample. This result is in fair agreement with our own experiments.

We are indebted to Dr. Thomas Stevenson for permission to use results obtained in his laboratory.

NOTE.—We are indebted to Mr. A. H. Allen for informing us, in a private communication, that P. Chabot (*Compt. rend.*, cxi., 231, and *Journal of the Chemical Society*, Abstracts, 1890, 1427) had previously determined the rotatory power of camphor when dissolved in various oils. A 20 per cent. solution in olive-oil gave the value $+55° 12'$, and this also agrees fairly with our own results.

ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

FOODS AND DRUGS ANALYSIS.

Foreign Colouring Matter in Milk. **A. E. Leach.** (*Journ. Amer. Chem. Soc.*, vol. xxii. [4], pp. 207-210.)—To detect foreign colouring matters the author curdles 150 c.c. of the suspected milk by the aid of heat and acetic acid, the curd being collected in a lump by means of a stirring-rod, or else separated by straining through a sieve. The whole of any annatto or aniline-orange and a portion of any caramel colouring matter employed will be retained by the curd, which is next broken up and treated with ether for the extraction of fat and annatto. The residual curd will be

white if the milk has not been artificially coloured, or if only annatto has been used; whereas caramel will leave a brown curd, and aniline-orange a more or less bright orange tinge.

Annatto can be separated from the ethereal extract by evaporating the latter, making the residue alkaline with sodium hydroxide and pouring it on a wet filter, which will absorb annatto and acquire a fairly permanent orange tint, turning to pink in presence of stannous chloride.

Caramel may be regarded as present if the coloured curd from which the fat has been extracted yields a deep blue coloration on agitation with concentrated hydrochloric acid, such as the white fat-free curd of an uncoloured milk would give,* and may then be confirmed by any of the usual tests on a fresh sample of the milk. When aniline-orange has been employed the acid treatment develops a pink coloration immediately. C. S.

Artificial Cooking Fat and its Digestibility in Comparison with Lard. H. Lührig. (*Zeit. für Untersuch. der Nahr. und Genussmittel*, 1900, iii., 73-87.)—This paper is a further instalment of the author's researches upon the digestibility of various fats (*cf.* ANALYST, 1899, xxiv., 237 and 283). Two preparations of cooking-fat were examined and compared as regards digestibility with lard. Preparation I. consisted of tallow and cotton-seed oil; Preparation II. of tallow, cotton-seed oil and lard, both being productions of a well-known margarine factory. The methods pursued were mainly the same as described in the author's previous communications. The following coefficients of digestibility were obtained: for lard, 96·36; for Preparation I., 96·09; for Preparation II., 96·47. H. H. B. S.

The Detection of "Vinoline" and Bordeaux Red in Wines. A. Casali. (*Staz. Speriment. Ital.*, 1900, xxxiii., 183-189.)—In the author's experience the artificial coloration of Italian wines is at the present time a very general practice, those from the central provinces being chiefly coloured with "vinoline," and those from the south with Bordeaux red.

The name "vinoline" is not applied to a single colouring matter, but usually denotes a mixture of different coal-tar reds. In Italian wines the author has found it to consist to a large extent of salts of rosaniline, such as the hydrochloride, acetate, nitrate, and (more rarely) the sulphate. In order to detect these, 100 c.c. of the wine are rendered strongly alkaline with ammonium hydroxide, and after the addition of a few drops of a solution of sodium hydroxide, shaken for a long time with 30 c.c. of chloroform. The solution of the colouring matter in chloroform becomes red on acidification, and when allowed to spontaneously evaporate leaves a scarlet residue.

Wines coloured with Bordeaux red (azo-naphthyl-naphtol-sulphonic acid) differ from those coloured with "vinoline" in giving a turbidity on the addition of hydrochloric acid. To detect this, 50 to 100 c.c. of the suspected wine are treated with 10 to 20 c.c. of a 10 per cent. solution of barium chloride, and sufficient potassium bisulphate

* In order to obtain this reaction, which is hastened by gentle heat, it is necessarythat the curd should be thoroughly freed from fat.

solution to precipitate the barium salt. When the precipitate has subsided, the supernatant liquid is decanted, and after the addition of 3 to 6 c.c. of hydrochloric acid and sufficient water to bring it to its original volume, it is boiled for 5 to 10 minutes with a few fibres of fat-free wool.

In the presence of Bordeaux red the wool is dyed a bright-red colour, which does not alter on adding ammonium hydroxide. In certain cases the colour appears to be carried down with the precipitate, and the wool, when boiled with the deposit and washed, assumes, on treatment with alkali, a green tint, which changes to red on again washing with water.

"Vinoline" colours which are not in the form of salts do not behave like the dyes mentioned above, but can be readily extracted from the wine by means of hot amyl alcohol. This extraction can be made even when 9 to 11 per cent. of alcohol are present and in the presence of acetic or hydrochloric acid. The wool dyed red in the amyl alcohol solution becomes green on treatment with ammonium hydroxide, and again red when washed with water. When dried and digested with dilute hydrochloric acid it colours the liquid violet-red—a property which distinguishes it from wool dyed with Bordeaux red. C. A. M.

Cider Vinegar; its Solids and Ash. R. E. Doolittle and W. H. Hess. (*Journ. Amer. Chem. Soc.*, vol. xx., [4], pp. 218-220.)—Two samples of imitation cider vinegar containing the statutory percentages of solids and ash were found on examination to furnish an evaporation residue resembling molasses, and of sharp acid odour, whereas the solids from the genuine vinegar are light and foamy in appearance, with an aroma of baked apples. The following results were obtained on analysis:

	Pure Cider Vinegar.	Apple Pomace Vinegar.	Suspected Vinegar. 1	Suspected Vinegar. 2	Boiled Cider.
	Per cent.	Per cent.	Per cent.	Per cent.	Per cent.
Solids:					
Reducing sugar, before inversion	0-14	0	42·88	56·82	61·12
Reducing sugar, after inversion	0-10	0	33·36	57·04	66·45
Polarization, before inversion	0°	0°	+ 40·7°	- 31·14°	- 17·9°
Polarization, after inversion	0°	0°	+ 38·8°	- 34·25°	- 25·0°
Ash constituents:	Per cent.	Per cent.	Per cent.	Per cent.	Per cent.
CaO	3·4-8·21	4·73	4·70	37·95	
MgO	1·88-3·44	4·12	2·00	2·22	
K_2O	46·33-65·64	37·00	nil	7·84	
Na_2O	trace	trace	49·71	trace	
SO_3	4·66-16·29	34·78	27·04	12·74	
P_2O_5	3·29-6·66	9·66	0·005	1·82	
Fe_2O_3	trace	trace	trace	1·60	
CO_2, loss, etc.	40·44-0	9·64	16·54	35·83	

The suspected sample No. 1 was therefore compounded of dilute acetic acid, glucose and soda ash; No. 2 of acetic acid, boiled cider, and lime. C. S.

The Detection of Foreign Colouring Matters in Preserved Tomatoes. G. Halphen. (*Journ. Pharm. Chim.*, 1900, xi., 169-172.)

1. *Coal-tar Colours.*—The usual method of testing for these consists in mixing the tomato-pulp with dilute ammonium hydroxide, extracting it with amylic alcohol, and evaporating the extract in the presence of undyed silk. In the author's experience this has not proved satisfactory, partly on account of the readiness with which certain colouring matters become attached to the vegetable fibres, and partly because the particles of tomato prevent the solvent from coming into contact with the colour.

He has obtained better results by extracting the desiccated pulp with ordinary alcohol from an acid medium. An addition of an equal volume of sand or precipitated silica is advantageous during the drying, which in certain cases should take place at the ordinary temperature in order to prevent the possible decomposition of unstable azo-compounds. In very many cases, however, the mass may be dried on the water-bath.

The dried substance is pulverized, transferred to a flask, and covered with glacial acetic acid. The mass is well stirred and the flask closed, and left for ten minutes. The liquid is then mixed with twice its volume of alcohol (90 per cent.), and again allowed to stand. After ten minutes it is transferred to a filter, and the filtrate collected in a 250 to 300 c.c. flask. After the addition of ten times its volume of water, a small knot of flossy silk is introduced, and the liquid boiled for at least fifteen minutes. In the absence of coal-tar colours the silk becomes slightly yellow or brown, without any trace of red or rose; but in the presence of these dyes it assumes a rose-red or salmon colour.

2. *Cochineal.*—According to the author, this is always used in the form of a lake, and as these lakes are often not attacked by glacial acetic acid, it is frequently possible to detect the dyes in the residue left on the filter in testing for coal-tar colours. It is preferable, however, to use a fresh portion of the tomato, which is mixed with silica and dried on the water-bath.

The dried residue is thoroughly mixed with pure hydrochloric acid (specific gravity 1·16 to 1·17), and after being left for ten minutes the paste is shaken with twice its volume of 90 per cent. alcohol, allowed to stand for ten minutes, and filtered.

The filtrate is mixed with at least ten times its volume of water, and is shaken with sufficient amylic alcohol to form a layer of not less than 5 c.c. when the liquids separate. The lower aqueous layer is withdrawn, leaving the carminic acid and part of the natural colouring matter of the tomato in the amylic alcohol.

In order to separate these, from 1 to 1·5 times the volume of carbon bisulphide and 4 to 5 times the volume of water are added to the amylic alcohol, and the mixture shaken with a circular motion. After standing, the lower layer is removed and the upper aqueous layer filtered. When cochineal has been used the filtrate will have a rose colour, with more or less of a yellow shade, and on shaking with 2 or 3 c.c. of amylic alcohol the colouring matter can be extracted, forming a yellowish-red solution. The amylic alcohol solution is tested with a drop of a concentrated solution of uranium acetate, and on shaking gives a characteristic green coloration.

C. A. M.

The Occurrence and Estimation of Tin in Preserved Meat. F. Wirthle. (*Chem. Zeit.*, 1900, xxiv., 263.)—The author has recently had the opportunity of investigating some samples of tinned meat, one, two, three, and four years old, which had been preserved in vessels of tin-plate with folded seams, solder not being used. He found that the proportion of tin in the food slightly increased during the time of keeping after the second year, but between the first and second year there was a minute falling off in metallic contamination. He also corroborated Günther's statement that the amount of tin in the meat itself is about two or three times as much as in the liquor. His maximum figures were obtained on a sample four years old, where the meat gave 0·0094 per cent., while another specimen of beef of the same age gave 0·0082 per cent. in the meat and 0·0028 per cent. in the juice. A sample of beef five years old, on the other hand, contained 0·0325 per cent. of tin, and the liquor 0·014 per cent., the inner surface of the vessel being completely destroyed. Corrosion of the can occurs mainly where it comes in contact with the fat; and the white deposit noticed in bad cases consists of basic stannous chloride.

For the determination, 120 grammes of meat (or liquor and fat) are heated in a porcelain basin over an asbestos plate with 5 c.c. of strong sulphuric acid; the mixture is stirred at intervals, and more acid (altogether 15 or 20 c.c.) is dropped in gradually, until a porous carbonaceous mass is obtained. This is powdered and rinsed with dry sodium carbonate into a porcelain crucible, more sodium carbonate and some nitrate are added, and the whole is fused. The melt is extracted with water and a current of carbon dioxide is passed through the liquid. When clear, the precipitate is collected, washed, dried, and ignited, then heated in the covered crucible with potassium cyanide to a dull-red heat. The mass is treated with water, the metallic iron and tin filtered off, and dissolved in warm hydrochloric acid. The tin is precipitated with sulphuretted hydrogen, collected, washed with sulphuretted hydrogen-water containing some ammonium nitrate solution, dried and ignited with the paper to constant weight. F. H. L.

ORGANIC ANALYSIS.

The Detection of Benzene in Alcohol. G. Halphen. (*Journ. Pharm. Chim.*, 1900, xi., 373-375.)—As it has been proposed in France to denature alcohol for commercial purposes by the addition of benzene oils, the question has arisen as to whether it is possible to detect the presence of benzene in the alcohol recovered from such denatured products.

The author finds that the following method is extremely sensitive and gives good results. It consists in isolating the hydrocarbons and converting them into diazo-compounds, which combine with naphthols to form coloured compounds.

From 10 to 20 c.c. of the spirit under examination are shaken with 5 to 10 c.c. of carbon bisulphide in a separating funnel, and after the addition of ten times the volume of sodium chloride solution (about 20 per cent.), the funnel is inverted several times and allowed to stand. The lower layer of carbon bisulphide is withdrawn, and the hydrocarbons nitrated by the addition of 0·5 to 1 c.c. of a mixture of 10 c.c. of Nordhausen sulphuric acid and 1 c.c. of fuming nitric acid, the liquids being stirred

together for two or three minutes. The supernatant carbon bisulphide is then decanted, 5 c.c. of water added, the rise in temperature produced accelerating the separation of the last portions.

The acid layer is diluted with 15 to 20 c.c. of water and shaken with 4 or 5 c.c. of sulphuric ether, in order to remove the nitro-compounds, which are nearly insoluble in water.

The residue left on evaporating this extract is treated with 20 c.c. of water acidulated with hydrochloric acid (5 to 10 per cent.), then with one drop of a solution of platinic chloride, and 0·2 to 0·3 gramme of zinc dust, and the evolution of hydrogen continued for at least ten minutes.

The liquid, which should still be acid, is filtered, and when cold is treated with 4 or 5 drops of a 10 per cent. solution of sodium nitrite, and after ten seconds the excess of acid neutralized by a slight excess of sodium carbonate.

The solution with the zinc carbonate in suspension is then tested with a few drops of an alkaline solution of α-naphthol, which, in the presence of nitrobenzene, produces an orange-red coloration.

Similar colorations are also obtained with the nitro-compounds of the higher homologues of benzene. C. A. M.

A Comparison of some Formaldehyde Tests. B. M. Pilhashy. (*Journ. Amer. Chem. Soc.*, vol. xxii. [3], pp. 132-134.)—The author finds that the following reagents recommended for the detection of formaldehyde, viz., Schiff's (fuchsine and sulphurous acid), phenol and sulphuric acid, diazobenzene-sulphonic acid, and Nessler's solution, also give reactions with other aldehydes, whilst the coloration produced by dimethylaniline and sulphuric acid (Trillat) is due to the dimethylaniline alone. Lebbin's test he finds unreliable below the limit of 1 : 200,000, whilst the limit of delicacy of the morphine hydrochloride test is 1 : 1000. On the other hand, phenylhydrazine hydrochloride forms the best test, a decided green coloration being developed in half a minute in a 1 : 100,000 solution, and in ten minutes with a 1 : 250,000 solution. For the same reagent in conjunction with sodium nitrocyanide and concentrated sodium hydroxide the limit of sensitiveness seems to be about 1 : 1,000,000. C. S.

Two Deceptive Reactions. C. Amthor. (*Zeit. für Untersuch. der Nahr. und Genussmittel*, 1900, iii., 233-235.)—*Detection of Formaldehyde.*—The author has studied the colour reaction with hydrochloric acid, which Leonard and Smith supposed to be indicative of milk containing formalin, but which Fisher pointed out was also obtained with milk containing boric acid (ANALYST, xxiv., 86). He finds that the reaction is neither characteristic of formaldehyde nor of boric acid, but is equally well produced when both bodies are absent. He ascribes it to the presence of albuminous bodies in conjunction with sugar (cane-sugar, milk-sugar, dextrose, etc.). With sour milk, owing to the small quantity of sugar remaining therein, the reaction is weak, but it becomes immediately stronger on the addition of a little milk-sugar.

The reaction can also be produced with meat or egg-albumin, but here also the addition of a little sugar is necessary to produce a strong reaction.

Detection of Sesame Oil.—The author found that the reaction above referred to was frequently obtained with butter, owing to the presence of small quantities of casein and milk-sugar, and that when only slight it could be intensified by the addition of milk-sugar. Further, that the same effect was produced when furfurol was substituted for sugar. The fact is of importance in connection with the detection of sesame oil in butter by means of furfurol, since unless the casein be previously completely removed by filtration or other means, the reaction may be obtained in the complete absence of sesame oil. H. H. B. S.

Note on the Estimation of Formic Acid in the Presence of Acetic Acid. F. Sparre. (*Zeit. anal. Chem.*, 1900, xxxix., 105, 106.)—The method given by Leys (*Zeit. anal. Chem.*, xxxviii., 677), which consists in heating the liquid with mercuric oxide, suffers from the drawbacks that the flask must be allowed to stand overnight, and that the precipitate must be filtered, washed, dissolved in nitric acid, precipitated with sodium chloride, and estimated gravimetrically.

In the author's experience, the method of Portes and Ruyssen is less tedious and equally accurate. In this 5 grammes of sodium acetate, 25 c.c. of a 1 per cent. solution of the liquid under examination, and 200 c.c. of a 4·5 per cent. solution of mercuric chloride are heated in a flask for one to one and a half hours on the water-bath, and then diluted to 500 c.c. and filtered. The amount of formic acid is then calculated from the number of c.c. of the liquid required to produce a red coloration with 1 gramme of potassium iodide. C. A. M.

A New Reagent for Phenolic Bodies. G. Candussio. (*Chem. Zeit.*, 1900, xxiv., 299.)—This reagent consists of a 1 per cent. solution of potassium ferricyanide, containing 10 to 20 per cent. of ammonia; and its effect on various phenols, etc., is shown in the paper, side by side with that of a 10 per cent. solution of ferric chloride. The tests are carried out in three ways: by adding the reagent drop by drop to the phenol; by dropping the phenol into an excess of the reagent; and by making the phenolic solution alkaline with sodium hydroxide, and then adding the reagent. Potassium ferricyanide exhibits several advantages over ferric chloride; and in particular it can be used on an alkaline or faintly acid liquid without previous neutralization, so that in the examination of essential oils, or in physiological and pathological work, it is distinctly preferable. Reducing bodies, such as sodium phosphite and hypophosphite, formaldehyde, and especially sodium sulphite, destroy the reactions, precisely as happens with ferric chloride. F. H. L.

The Theory of Saponification. J. Lewkowitsch. (*Berichte*, 1900, xxxiii., 89-99.)—Geitel came to the conclusion that during the saponification of fats and oils mono- and di-glycerides are produced as intermediate products, but Henriques (ANALYST, 1898, 290) was unable to obtain any confirmatory evidence of this. The author has therefore made a large number of experiments in order to settle the

question. In these, several pounds of tallow and of cotton-seed oil were saponified with sodium or calcium hydroxides in the same way as in the ordinary manufacturing process. From time to time samples were withdrawn, treated with hydrochloric acid, and washed and dried. The acetyl value and the Hehner and saponification values of the acetylated product were determined, whilst the amount of free acid calculated from the acid value showed to what extent the saponification had progressed.

The line of reasoning followed was, that if the saponification only produced glycerin and fatty acids, the acetyl value of the partially saponified fat should not exceed that of the original fat; but if, on the other hand, lower glycerides were formed as intermediate products, these having a definite acetyl value would cause an increase in the acetyl value of the samples (*cf.* ANALYST, xxiv., 326).

Confirmatory evidence would also be obtained from the Hehner and saponification value of the acetylated product, which would show great variations in the presence of mono- or di-glycerides.

The experiments proved that the acetyl values rose and fell with the progress of the saponification, whilst at the same time the Hehner and saponification values of the acetylated products showed a corresponding variation. The author, therefore, has arrived at the conclusion that there can be no doubt that the saponification process takes place in stages, and that it must be regarded, not as a tetramolecular change, but as a bimolecular reaction in agreement with the views of Geitel.

As a typical example, the following results of one of the experiments may be quoted:

SAPONIFICATION OF COTTON-SEED OIL WITH CALCIUM HYDROXIDE.

		ACETYLATED PRODUCT.		
	Acid Value.	Acetyl Value by Filtration Process.	Hehner Value.	Saponification Value.
Original oil acetylated ...	0·0	11·7	93·5	—
Partially saponified oil:				
1.	0·5	14·9	94·5	206·3
2.	0·6	20·0	92·84	209·2
3.	16·0	43·15	92·0	230·1
4.	17·6	59·2	89·1	240·0
5.	19·9	28·3	92·35	215·3
6.	53·4	24·9	93·8	214·8
7.	73·2	32·4	93·6	223·4
Fatty acids obtained by saponification with alcoholic KOH, acetylated	199·45	13·8	99·4	216·4

Of this experiment the author remarks that the fairly rapid rise of the acetyl value, with at the same time the slower rise of the acid value, indicates the predominance of the first or first two steps in the saponification, whilst the subsequent rapid decrease in the acetyl value points to the decomposition of the monoglycerides predominating.

It is pointed out that in such experiments as these only the mean values of the concurring phases of the saponification are determined, since simultaneously a molecule of diglyceride may be converted into monoglyceride and fatty acids, or a molecule of monoglyceride into fatty acids and glycerin, while a molecule of triglyceride is only passing through the first stage.

In one of the experiments the saponification proceeded with such rapidity, that but for the fact that a higher acetyl value was obtained with one of the first samples, the results might have appeared to support the older view of saponification.

C. A. M.

Volumetric Estimation of Fatty Acids. J. Swoboda. (*Chem. Zeit.*, 1900, xxiv., 285.)—Standard alcoholic solutions of alkali hydroxides have the disadvantage of not retaining their strength for long; whereas in the titration of fatty acids aqueous alkalis are inconvenient, because of the precipitation of the fatty matter. The author suggests that fatty acids should be dissolved for titration in a mixture of 1 part of absolute ethylic alcohol and 2 parts of amylic alcohol, to which liquid an aqueous alkali may be added without causing separation, and in which the colour of the indicator is perfectly distinct. F. H. L.

Mixtures of Maize Oil with Cotton Oil. G. Morpurgo and A. Götzl. (*Oesterr. Chem. Zeit.*, 1900, iii., 53.)—Inasmuch as cotton oils with a low solidifying point are greatly sought after and fetch a high price, an addition of maize oil is quite possible, although its price is higher than ordinary cotton oil. The authors give a table of the constants of both oils as obtained from the examination in duplicate of four separate brands of American cotton and two brands of maize oil, the figures "agreeing fairly well" (*sic; cf.*, however, ANALYST, 1899, xxiv., 16) with those already quoted by De Negri and others. The most prominent differences between the oils are found in their iodine values and the melting-point of their fatty acids, and the following table represents these constants in the case of the genuine oils and in various mixtures thereof:

	Iodine Value.	Melting-point of Fatty Acids.
Cotton oil, mean figures*	106·47	36-37°
4 parts cotton oil + 1 part maize oil ...	107·40	36-37°
3 ,, ,, + 1 ,, ,, ...	110·80	35-36°
2 ,, ,, + 1 ,, ,, ...	112·05	34-34·5°
1 part ,, + 1 ,, ,, ...	115·70	31-32°
1 ,, ,, + 2 parts ,, ...	118·30	29-30°
Maize oil, sample No. 1	147·60	18-20°
,, ,, No. 2	124·50	18-20°

Maize oil does not give the Becchi reaction, so that additions of cotton oil can be detected in this manner. Taste and smell cannot be relied upon: hot-pressed cotton oil is much like roasted maize oil; sometimes, however, the latter is practically odourless. The acid value of pure commercial cotton oil is generally under 1, and it does not increase much when the oil becomes rancid; maize oil oxidises more readily, so that an excess of free acid points to the presence of maize oil in cotton oil.

F. H. L.

* Extreme figures, 104·45 and 107·61.

The Detection of Cotton-seed Oil by the Pentosan Reaction. B. A. van Kettel. (*Monit. Scientif.*, 1900, xiv., 154-156.)—In a former communication (*Ber. Nederl. Maatschap. Pharm.*, October, 1897) the author described a test based upon the fact that vegetable oils, and notably cotton-seed oil, contain traces of pentosans, whilst animal oils are free from such substances. Ten grammes of the oil are heated with a few c.c. of hydrochloric acid (specific gravity 1·06) until permeated with the acid vapours. The tube is then left for 10 minutes for the liquids to separate, and on now adding a solution of phloroglucinol hydrochloride, a red ring is formed at the juncture of the layers, in the presence of pentosans.

Among the oils which do not give the pentosan reaction are cacao oil, earthnut oil, croton oil, rape-seed oil, and linseed oil. Two specimens of pure olive oil examined by the author also gave negative results.

In the present paper the results of this test are compared with those of Halphen's and Cavalli's reactions. The latter test consists in shaking the oil with a mixture of 2 grammes of resorcin, 20 c.c. of water, and 15 c.c. of sulphuric acid, a red coloration changing to blue being given by cotton-seed oil.

Six specimens of cotton-seed oil of different origin and a specimen of cotton "stearin" were tested. With Halphen's reagent all gave a red coloration with the exception of a crude yellow Egyptian oil. With Cavalli's reagent only two of the oils gave a pronounced reaction, and one a faint coloration. With the author's pentosan reagent five of the samples, including the Egyptian oil, gave a marked coloration, and the other two a faint reaction.

The crude Egyptian oil was treated with animal charcoal and the filtrate tested with Halphen's reagent, but as the red coloration was still not obtained, the conclusion was arrived at that this oil did not contain the substance producing the reaction.

In view of the results of these experiments, Cavalli's test was regarded as unreliable.

The author found that his own reagent was capable of detecting 10 per cent., and sometimes 5 per cent. of cotton-seed oil in earthnut oil.

Holde and Pelgry have shown (ANALYST, xxiv., 214) that cotton-seed oil, after being heated at 220-225° C., no longer reacts with Halphen's reagent. The author has tested his pentosan reagent on the six oils mentioned above, after heating them for 30 to 40 minutes at about 250° C. Only two gave a coloration with Halphen's reagent, whilst with the author's reagent two gave negative results, four faint reactions, and one a pronounced coloration. C. A. M.

Separation of the Constituents of Rancid Fats and Oils. J. Nagel. (*Amer. Chem. Journ.*, 1900, xxiii., 173-176.)—The following classes of compounds were identified by the author in his investigation on the rancidity of fats and oils: (1) Free fatty acids (saturated and unsaturated). (2) Hydroxy-fatty acids. (3) Lactones and anhydrides of fatty acids. (4) Alcohols, such as butylic, amylic, and caprylic alcohol. (5) Esters of saturated, unsaturated, and hydroxy-fatty acids, with higher and sometimes also polybasic alcohols. (6) Aldehydes (saturated and unsaturated). (7) Acetals and (8) Terpenes.

I. *Separation of Saturated, Unsaturated, and Hydroxy Acids.*—These were neutralized with a solution of soluble glass instead of with alkalies, with the result that emulsions were avoided, the silicic acid liberated in the reaction drawing the alkali salts mechanically to the bottom.

II. *Separation of Lactones.*—Those lactones which were volatile with steam were removed simultaneously with terpenes by distillation, as in VI. Non-volatile lactones were converted into salts of hydroxy-fatty acids by boiling the oil, from which the free fatty acids had been removed, with a concentrated solution of an alkali. The resulting insoluble salts were separated by filtration.

III. *Alcohols and Esters*, when volatile with steam, were removed as described under VI.

IV. *Separation of Aldehydes.*—When volatile, these were removed by distillation. To separate the non-volatile aldehydes 4 volumes of the oil were heated for an hour with 1 volume of a concentrated solution of sodium bisulphite, and the aqueous layer separated on cooling. The compounds of the aldehydes with sodium bisulphite being slightly soluble in the solution of bisulphite, were removed together with the solution.

V. *Separation of Acetals.*—Where these were not volatile with steam, it was found best to decompose them into alcohols and aldehydes by heating the fat with dilute sulphuric acid. The resulting compounds were removed, as in IV. or VI.

VI. *Separation of Terpenes.*—These were removed by distillation, being all volatile with water-vapour. In order to prevent further changes in the oil by the prolonged action of steam at 100° C. and of atmospheric oxygen, the steam was either passed through the oil together with a neutral gas, or was gradually superheated (100° to 170° C.) and accompanied by an inert gas. In fractionating with superheated steam, the most volatile substances passed over before the more highly-heated steam, which would have decomposed them, was introduced. The oil was subsequently cooled *in vacuo* or in an atmosphere of an inert gas, such as hydrogen or carbon dioxide.

In conclusion, the author describes a method of practically applying the results of his investigation to the purification of rancid fats and oils on a large scale.

C. A. M.

Macassar Oil. **J. J. A. Wijs.** (*Chem. Rev. Fett. u. Harz-Ind.*, vol. vii. [3], pp. 46, 47.)—A sample of the butter-like fat obtained from the seeds of *Schleichera trijuga Willd.*, by extraction with benzene, furnished the following constants: Melting-point (Le Sueur and Crossley's method) of the fat, 22° C.; of the fatty acids, 52° to 54° C.; Hehner value, 91·55; saponification value (Henriques' cold method), 215·3; iodine value (Wijs' method) of the fat, 55·0; of the fatty acids, 58·9; Reichert-Meissl value, 9; acid value of the fat, 19·2; of the fatty acids, 191·2 to 192; percentage of unsaponifiable matter, 3·12 per cent.

The seeds yielded 40 per cent. of kernel, containing 70·5 per cent. of fat, 3·5 per cent. water, and 12 per cent. protein. The volatile acids, examined by the Duclaux method, were found to consist of acetic acid with a small quantity of a higher acid: probably butyric, by the odour of the ester furnished on treating the

distillate with alcohol and sulphuric acid. Of the non-volatile acids, 45 per cent. consisted of solid fatty acids and 55 per cent. of liquid acids, having the iodine value 103·2. C. S.

The Composition of Japan Wax. A. C. Geitel and G. van der Want. (*Journ. prak. Chem.*, 1900, lxi., 151-156.)—Considerable variations were found in the constants of four specimens of the commercial wax examined by the authors. The acid value fluctuated between 21·7 and 32·6; the saponification value between 217·5 and 237·5; the iodine value between 8·3 and 8·5; and the amount of unsaponifiable matter between 1·48 and 1·63 per cent. In two different samples 90·62 and 90·66 per cent. of insoluble fatty acids, and 4·66 and 5·96 per cent. of soluble fatty acids, were respectively found.

The soluble fatty acids had the consistency of lard; their acid value was 345·2, and their mean molecular weight 162. In the authors' opinion they consisted of a mixture of fatty acids, produced by the action of the oxidizing agents employed to bleach the wax.

The insoluble fatty acids had an acid value of 212·2 to 215·7, and an iodine value of 10·6. By fractional precipitation with magnesium acetate they were separated into palmitic acid, oleic acid contaminated with unsaponifiable matter, and a fatty acid melting at 117° C., to which the authors gave the name of *Japanic acid.*

This acid, when purified by recrystallization from alcohol or chloroform, melted at 117·7° to 117·9° C. It was found to be saturated, and to contain no hydroxyl groups. It could be dried without loss at 115° to 120° C. Its molecular equivalent, as found by titration, was 186·2, but by the boiling-point method its molecular weight was found to be 365. The conclusion finally arrived at was that it belonged to the succinic acid series, and had the formula $C_{20}H_{40}(COOH)_2$.

By recrystallizing the wax from hot benzene a hard, wax-like substance, melting at 43° to 45° C., and with an ester value of 252·6, was eventually obtained. As a glyceride containing one molecule of *Japanic acid* and one molecule of palmitic acid would have an ester value of 253·4, the authors considered it highly probable that this acid is present in Japan wax in the form of a mixed glyceride. A reference is made to the abnormal behaviour of the fatty acids of Japan wax when dissolved in a saturated alcoholic solution of stearic acid (ANALYST, xxi., 330). C. A. M.

Notes on Commercial Glycerin. H. Struve. (*Zeit. anal. Chem.*, 1900, xxxix., 95-99.)—The author gives a detailed account of a number of experiments, from which he has arrived at the following conclusions:

1. The purest glycerin in the market contains from 6·02 to 8 per cent. of water.

2. Commercial glycerin does not lose the whole of its water when dried *in vacuo*, but retains 1·52 per cent.

3. The most anhydrous glycerin is hygroscopic, and absorbs moisture from the atmosphere up to 17·46 per cent.

4. Traces of glycerin are volatile in a current of steam. In one experiment the loss on distillation amounted to 0·379 per cent. C. A. M.

Some Properties of Rosin. **A. Smetham and F. R. Dodd.** (*Jour. Soc. Chem. Ind.*, 1900, xix., 101.)—The authors call attention to the fact that the constants of colophony acids are not the same as those of the rosins from which they have been derived; therefore, in the analysis of soaps, especially those containing much rosin, it is not possible to calculate accurately the proportion of colophony used by the manufacturer by comparing the analytical data with the figures published for ordinary rosin. The annexed table gives, firstly, some constants of various brands of commercial rosin, and secondly the similar constants of the same materials after saponification, decomposition of the soaps, and removal of the resinous matters by means of ether.* The iodine values were obtained with the Hübl solution at eighteen hours, employing an excess of 300 per cent. of iodine.

	Brands.				
	"Wite."	"Nemo."	"W. W. Zante."	"N. Xeres."	"W. G. Wax."
Original rosin:					
Acid value	160·8	162·0	154·5	157·3	160·1
Saponification value ...	177·6	176·4	174·3	174·3	177·3
Iodine value	184·7	181·0	158·5	168·4	165·9
Recovered resin:					
Acid value	163·9	163·9	159·1	159·1	161·0
Saponification value ...	176·4	175·3	167·5	165·5	169·5
Iodine value	175·5	166·8	154·9	149·9	153·6

The authors agree with Lewkowitsch that the effect of the Wijs and Hübl iodine solutions on fats and oils is fairly concordant; but on rosin or resin acids it is very different. One sample of rosin gave a Hübl value of 156·7, and a Wijs value of 250·6 at eighteen hours. The widely different results obtained by the two methods would suggest that chlorine compounds are formed, and this seems to be borne out by the immediate change in the colour of the Wijs solution on coming into contact with the rosin, whereas in the case of tallow acids no such change occurs. The authors therefore conclude that the iodine process as at present worked is of doubtful utility in determining the nature of the fatty matters in a mixture of these with resin acids.

F. H. L.

Essential Oil of Chrysanthemum. **G. Perrier.** (*Bull. Soc. Chim.*, 1900, xxiii., 216, 217.)—The green leaves of *Chrysanthemum japonicum*, or autumn chrysanthemum, furnish, on distillation with water, a green essential oil. The yield (about 16 per cent.) varies with the different species of the plant, the maximum amount being obtained just before the flowers begin to fall.

The essential oil is a liquid of an oily consistency, with an odour recalling that of peppermint and that of chamomile. It begins to boil at 160° C., and has a specific gravity of 0·932 at 15° C., and a refractive index of 1·4931 at 18° C. It is soluble in 10 volumes of 95 per cent. alcohol, but is nearly insoluble in 70 per cent. alcohol.

* These latter figures, however, can scarcely be considered to represent the constants of colophony acids, for the unsaponifiable matter of the rosin, except that "removed in [aqueous] solution or by volatilization in the process of drying," was not extracted. The abstractor has usually found the proportion of unsaponifiable matter in the low-grade rosin "B.C." to be about 7 per cent.—F. H. L.

When cooled to −15° C., it deposits a small quantity of a solid amorphous substance, probably a paraffin; at −24° C. it becomes viscid, and solidifies when cooled in a mixture of ether and solid carbon dioxide. It combines partially with sodium bisulphite, is acid to litmus, and has a saponification value of 8·61. The alkaline salt formed in the saponification yields, when treated with hydrochloric acid, a solid substance with an odour of angelic acid.

From a preliminary examination the author considers that chrysanthemum oil resembles the oil of Roman chamomile in composition. C. A. M.

Properties of some Essential Oils. Schimmel and Co.'s April Report. (*Chem. Zeit.*, 1900, xxiv., 304.)—*Oil of Basil.*—Two descriptions of this substance are now recognised. From the large-leaved variety of *Ocymum basilicum*, known locally as Selasih Mekah, between 0·18 and 0·32 per cent. of oil can be obtained by distillation, which has a specific gravity of 0·90 at 26° C., and a rotatory power of −30·5° to −36° in a 200-millimetre tube. The oil contains from 30 to 40 per cent. by volume of eugenol. From its neutral constituents a body of agreeable odour, boiling at 190° C., has been recovered, and is now under investigation. The other variety of *O. basilicum* yields about 0·2 per cent. of oil, smelling like fennel, with a specific gravity of 0·948 at 25° C. It boils mostly between 214° and 218° C., and consists largely of methyl-chavicol. On treatment with alcoholic potash, it gives anethol; and on oxidation, anisic acid.

Oil of Iris.—J. C. Stead has succeeded in obtaining from 5 parts of the usual solid or buttery material 1 part of a golden-yellow oil, with a fine odour of violets when highly diluted. It is soluble in all proportions in ether, alcohol, chloroform, petroleum spirit and benzene, and yields a clear mixture with 8 volumes of 70 per cent. alcohol. It is acid to litmus. Its optical values are $d_{15·5} = 0·9489$; $[\alpha]_D = -28·25°$. It solidifies at −5° C. It decomposes on distillation at atmospheric pressure, and contains no constituent separable by bisulphite; but by treatment with alkali before and after acetylation is shown to contain free acids and some alcoholic bodies.

Oil of Rosemary.—Recent investigations by Guerbet of pure santalol recovered from its phthalic acid compound throw doubt on the correctness of the hitherto accepted formula, $C_{15}H_{26}O$. On hydrolysing acetylated santalol, figures were obtained corresponding to a content of 103·5 per cent. of santalyl acetate, whence santalol must either have a smaller molecule (perhaps $C_{15}H_{22}O$), or it must be a mixture of two differently composed alcohols.

Oil of Wartara.—This is a new product, prepared by Schimmel from Bombay Wartara seeds, which closely resemble the fruit of *Xanthoxylum piperitum D. C.*, and probably also belong to the *Rutaceæ*. On distillation, about 2 per cent. of an oil smelling like coriander was obtained: specific gravity, 0·8714; rotatory power, $[\alpha]_D = +6° 31'$; saponification value, 27·1; solubility, 1 vol. in 1 vol. of 80 per cent. alcohol. By distillation at a pressure of 14 millimetres it was separated into six fractions, of which the first boiled at 175° to 176° at atmospheric pressure, and gave a large yield of a tetrabromide melting at 125°, showing it to be almost pure pentene. The fractions 3 to 5 were rectified, and finally an oil was recovered which exhibited the properties of *d*-linalol: specific gravity, 0·868; rotatory power, +14° 20′; boiling-

point, 78° at 14 millimetres. The identity of this oil with *d*-linalol, which has hitherto been found only in oil of coriander, was proved by its conversion into citral and into *l*-terpineol, etc. F. H. L.

On Certain Methods for the Determination of Cellulose. C. Beck. (*Zeit. für Untersuch. der Nahr. und Genussmittel*, 1900, iii., 158-164.)—The methods studied by the author were Lebbin's (*Arc. Hyg.*, 1897, xxviii., 212), König's (*Zeit. für Untersuch. der Nahr. und Genussmittel*, 1898, i., 3-16; ANALYST, 1898, xxiii., 47), and Henneberg's. Lebbin's method is briefly as follows: 3 to 5 grammes of the substance are first boiled with 100 c.c. of water for half an hour; 50 c.c. of 20 per cent. hydrogen peroxide are then added and the boiling continued for twenty minutes, during which 15 c.c. of a 5 per cent. ammonia solution are added in small quantities at a time. This is followed by another boiling for twenty minutes, after which the raw fibre is filtered off, washed with hot water, dried and weighed. König's method has already been published in this journal (*vide* abstract cited above). The old method of Henneberg (boiling successively with dilute sulphuric acid and potash solution) was studied in conjunction with the two newer processes for the sake of comparison. In all cases the amount of cellulose was arrived at by deducting the sum of the pentosans (determined by Tollens and Councler's method), the nuclein (N by Kjeldahl × 6·25), and the ash from the raw fibre. The most concordant results were obtained with Henneberg's method, and the author considers that for establishing a comparison between the finer sorts of flour, this method furnishes the most reliable data. Lebbin's method yielded the least concordant results. In connection with this method, the author tried the effect of removing, as far as possible, the starch and the albuminous bodies before boiling with the hydrogen peroxide. The results, however, were still unsatisfactory, and are only interesting in so far as they show the unsuitability of hydrogen peroxide for the purpose, even under conditions more favourable than obtain in the original method. The author does not consider Lebbin's method of any value at all, either scientific or practical. The results obtained by König's method were also very discordant, owing apparently to variations in the boiling-point of the liquid. It also seemed that some other substance must be left in the raw fibre in König's method, since the cellulose found in bran and flour by this method by deducting the nitrogenous substances, pentosans and ash from the raw fibre was greater than that obtained by Henneberg's method, notwithstanding that the cellulose is less attacked in Henneberg's method than it is in König's.

Remarks on the Foregoing Paper by J. König.—König remarks that the discordant nature of the results obtained by Beck with his method was probably due to his instructions regarding temperature not having been accurately followed. The temperature should be maintained within the limits of 131° and 133° C. This can easily be done by using glycerin of 1·229-1·230 specific gravity, working upon air-dried substances containing not more than 12 per cent. of water, and reducing the flame of the burner as soon as the fluid commences to boil. The use of a digester, however, is preferable to open boiling, and in this case the temperature should be maintained at 137° C. for one hour. Beck's opinion as regards the suitability of Henneberg's method for discriminating between fine flours is directly contrary to König's experience. He considers that Henneberg's method is less reliable with fine

flour, and rarely gives concordant results, on account of the difficulty of filtration. König remarks further, that his object, as stated in his paper, was to devise a method for separating as completely as possible the pentosans from the hexosans, and that no method yet proposed furnishes pure cellulose of the formula $n(C_6H_{10}O_5)$.

H. H. B. S.

Notes on the Estimation of Cellulose. C. Councler. (*Chem. Zeit.*, 1900, xxiv., 368.)—The author discusses four methods which have been proposed for this determination: (1) The Schulze-Henneberg process, (2) H. Müller's process, and (3) that recommended by G. Lange (ANALYST, 1895, xx., 283). Müller's process is carried out by extracting the material with a mixture of alcohol and benzene, and next with hot water; then removing from the residue certain "incrusting substances" by repeated alternate treatments with 0·4 per cent. bromine-water and 0·4 per cent. hot ammonia, filtering and washing the mass between each stage of the operation. Müller claims that four such treatments with bromine and ammonia are sufficient to yield pure cellulose; but Councler always finds more, sometimes twenty, alternations to be necessary. The method thus becomes exceedingly laborious, and Councler has endeavoured to shorten it by (4) previously heating the sample with calcium bisulphite solution (8° Beaumé) for four or eight hours at a temperature of 110° to 140° C. in a sealed tube, and then proceeding as before.

All these four processes show different results, (3) and (4) giving the lowest, but not identical figures; (3) is untrustworthy because, no true solvent for cellulose having yet been discovered, the substance dissolved by Lange's alkali and precipitated by his strength of acid is not cellulose; (4) is incorrect because cellulose appears to be attacked by bisulphite at temperatures of 100° C. and upwards; (1) and (2) seem to give better results, but are too complicated and tedious. An exact and simple method of estimating cellulose has therefore yet to be elaborated.

F. H. L.

On the Determination of Carbon and Hydrogen by Combustion in Oxygen, using Copper Oxide. C. F. Mabery and W. R. Clymer. (*Journ. Amer. Chem. Soc.*, vol. xxii., [4], pp. 213-218.)—In investigating the conditions necessary to insure accuracy in the combustion analysis of hydrocarbon oils the authors find that copper oxide is reliable when brought into a constant condition by blank trials. For the drying apparatus they recommend combustion-tubes 175 centimetres long, bent at an angle of about 105° at 30 centimetres from one end, one set being filled with broken glass and concentrated sulphuric acid, the other with glass and 40 per cent. potassium hydroxide solution.

Sulphuric acid forms the best absorbent for the water produced during combustion, and when used in small quantity (2 to 3 c.c.) retains but a very slight proportion of carbon dioxide. The tube employed has a bulb for collecting the greater part of the condensed water; the front limb of the U-tube is 10 centimetres high, and a lateral tube is attached for connection to the potash bulb. This drying-tube may be used for ten to twelve combustions before recharging with acid.

For carbonic acid absorption the Geissler bulb is the most convenient apparatus,

and no loss occurs, provided the 33⅓ per cent. potassium hydroxide be used for not more than four or five analyses. With the ordinary calcium chloride tube air may be safely passed at the rate of 500 c.c. per minute, and the freshly-fused, fine granular chloride may be used in a large number of analyses.

The combustion-tubes require a long time to bring them and the connections into good working order—expulsion of volatile impurities from the copper oxide, moisture from the tube, oxide, and rubber stoppers, etc. If the tube be filled with oxygen before inserting the substance, and the rate of volatilization be carefully controlled, any hydrocarbon may be burned in a 20-burner furnace. No advantage is gained by weighing the absorption-bulbs full of oxygen instead of air. C. S.

The Elementary Analysis of Organic Substances containing Nitrogen. F. G. Benedict. (*Amer. Chem. Journ.*, vol. xxiii., [4], pp. 334-352.)—To secure the reduction of the copper without adding unknown amounts of carbon dioxide and water to the materials to be weighed, the author recommends that such nitro-compounds as contain a deficiency of carbon and hydrogen in the molecule should be burned in admixture with a carbonaceous substance of known composition, the resulting products being then in a position to react upon and reduce the oxides of nitrogen present. Naphthalene, benzoic acid, and sucrose are highly suitable, but the first-named is difficult to obtain pure, whereas pure, dry sucrose can be readily obtained.

It is generally desirable, in charging the boat, to leave a free space of about 1 centimetre at the front end, which space is filled with the reducing agent (*e.g.*, sucrose), the rest being sprinkled over the substance. A space of 1 centimetre is left between the front end of the boat and the asbestos plug in the combustion-tube. The spiral in the front end of the tube is heated first, and then the copper oxide, before the reducing material in the boat is reached. The gases evolved by the sucrose reduce part of the adjacent copper oxide; and the final charring of the sucrose leaves an excess of carbon to aid in reducing the nitro-group. The conditions are different with benzoic acid, there being little or no carbon left, though the copper oxide is reduced for a distance of several centimetres. In calculating the results, allowance is made for the known weight of water and carbon dioxide formed by the reducing agents employed.

An admixture of three or four volumes of finely-powdered, dry silica increases the regularity of combustion, and appears to have a direct influence on the reduction of the oxides of nitrogen, which under these circumstances are found in much smaller quantities in the products of combustion. C. S.

A Colour Reaction given by the Products of the Digestion of Fibrin and Albumin with Papayotin. V. Harlay. (*Journ. Pharm. Chem.*, 1900, xi., 172-178.)—The products of the digestion of proteids with the juice of *Carica hastifolia* were found by the author to give with tyrosinase the same coloration as that given by the products of peptic digestion (ANALYST, xxiv., 272), but in a greater degree of purity. The green colour in the case of pepsin resembled that of chlorophyll, whilst the

papayotin green had more of a blue shade. This was shown to be due to the presence of other colouring matters which accompanied the pepsin green, and which on reduction with zinc and hydrochloric acid were destroyed first. In both digestions the main colours had the same spectrum and reactions, and were considered by the author to be identical. C. A. M.

A Method of distinguishing Indigo from other Blue Dyes in Woven Fabrics. F. H. van Leent. (*Zeit. anal. Chem.*, 1900, xxxix., 92-95.)—The threads of the fabric under examination are disintegrated into a loose wool, and a portion covered with pure melted phenol in a test-tube, which is then kept for an hour in a boiling water-bath, with occasional agitation. The blue solution which is obtained when indigo is present is poured off from the fibres, and the treatment with phenol repeated if required. The fibres are then washed two or three times with strong alcohol, and, after pressing and drying, a pure white wool is left.

Indigo red and another red colouring matter, possibly originating from the indigo, are simultaneously dissolved by the phenol. When the phenol solution is mixed with a large quantity of strong alcohol, the greater part of the indigo blue is precipitated, and the liquid shows the colour of the indigo red.

If the fibres be boiled with the phenol, they are strongly attacked by it, which, however, is not the case at the temperature of boiling water. Aniline and nitrobenzene extract but little indigo from dyed wool at 100° C., and are therefore unsuitable solvents for the test.

In experiments with wool dyed with other colouring matters, the author has so far found that, with the exception of indophenol, indigo is the only dye dissolved by phenol under the above conditions.

The results of his experiments are shown in the following table :

Pure indigo	Colour completely extracted; fibres white.
Indigo with sandal-wood	Indigo removed; red colouring matter left on the fibres.
Indigo and alizarin blue with chrome mordant	Indigo removed; fibres blue.
Indigo over alizarin	Indigo removed; fibres red.
Indigo and indophenol in mixed bath	Much colour removed; fibres still blue.
Indigo sulphonic acid	Only a little colour removed; fibres blue.
Brilliant sulphonazurine	Nothing removed; fibres unchanged.
Indulin	A little removed; fibres blue.
Alizarin indigo blue	Nothing removed; fibres unchanged.
Alizarin cyanin	Liquid coloured red; fibres remain blue.
Anthracene blue	Some red extracted; fibres unchanged.
Logwood with mordant of alum, chromium, and antimony ...	Unchanged.

Other dyes, such as palatine scarlet, cochineal, and anthracite black, also remained undissolved by the phenol. On testing a mixture of fibres dyed with pure indigo, with anthracene blue, and with indigo and sandal-wood, only the indigo dissolved, and, under the microscope, the different colourless, blue, and reddish brown fibres could be identified.

The test is not so suitable for cotton as for woollen fabrics, since indoïn is dissolved from cotton by the phenol as readily as indigo.

The author has not succeeded in quantitatively estimating the indigo by this method, chiefly on account of the difficulty of separating the fibres from the phenol solution without loss. C. A. M.

Notes on the Valuation of Tanning Materials. Paessler. (*Zeits. angew. Chem.*, 1900, 318.)—The author holds that if the directions laid down in the "Report of the First Congress of Leather-Trades Chemists" (H. Proctor and J. P. Parker) are followed exactly, the results obtained on analysing samples of tanning materials will become quite satisfactorily uniform and accurate. He also calls attention to the following points: It is necessary to take great care in selecting a small sample for analysis, even when the material has already been sampled from bulk. Many tanning materials contain a large number of foreign substances of different specific gravities, and so tend to separate into layers of different composition during agitation or transport. Especially with materials which are but imperfectly soluble in water, the particular grade of filter-paper employed is of considerable influence on the results. Schleicher and Schull's No. 602 "Extra Hard" is the best; in all cases the filtrate must be perfectly clear, for even the trace of suspended matter that passes a softer paper and renders the liquid only opalescent is taken up by the hide-powder and affects the figures of the analysis. The use of a hide-powder containing a certain proportion of filter-paper is distinctly advantageous; the cellulose prevents the liquid from rising up the walls of the filtering vessel, and also assists in the washing of the tanned hide-powder. Mehner and Stransky of Freiberg-i.-S., are now making a powder with 15 to 18 per cent. of filter-paper (instead of the 35 per cent. advised by Cerych), which behaves admirably. The hide-powder is best packed so that the filtrate passes at about 10 drops per minute; excessive tightness causes loss of tannins by dissolution of the soluble matters of the powder, but the errors are not serious. It is advisable, and in special cases important, that the temperature of the extract when being diluted to the desired volume, and of the liquid during filtration, should be maintained at the normal point, 17·5° C.; for the degree of solubility of some of the almost insoluble tanning constituents depends largely upon that temperature. F. H. L.

The Detection and Estimation of certain Vegetable Substances commonly used to adulterate Sumach. G. Scarlata. (*Staz. Speriment. Agrar.*, 1900, xxxiii., 168-171.)—Sicilian sumach should consist of the dried and powdered leaves of *Rhus coriaria*; but from the author's experience it appears to be widely adulterated with the leaves of other plants containing tannin, and notably with those of the *Tamarix africana* and of the lentiscus (*Pistacia lentiscus*).

Of the methods proposed to detect these adulterants, a microscopical examination of the powder, as recommended by Brizi, requires long experience, and is, hence, practically unused, whilst that of Spica (*Gazzetta*, 1897, 354) does not solve the problem.

The author's method is based on the fact that when finely-powdered sumach is

allowed to fall upon glycerin (specific gravity 1·26), which is then brought to the boiling-point and left for an hour, the sumach subsides completely, and the supernatant glycerin assumes a greenish-yellow tint.

If, however, the test be tried with finely-powdered lentiscus leaves, the powder is left on the surface of the glycerin, to which it imparts a wine-red colour; whilst in the case of the tamarix leaves the powder remains in suspension, and only falls after twenty-four hours. Here, too, the glycerin is coloured wine-red.

In his experiments on the quantitative determination of lentiscus in sumach, the author prepared a mixture containing 20 per cent. of the former. Five grammes of this mixture, previously passed through a fine-meshed sieve, were introduced into a large test-tube containing 110 c.c. of glycerin (specific gravity 1·26); the liquid was heated to the boiling-point, without shaking, and boiled for a few moments until there was a complete emulsion. It was then left for twenty-four hours, after which the supernatant glycerin, together with the powder on its surface, was removed by means of a pipette. The powder was collected on a weighed filter, washed with water until free from glycerin, dried in the water-oven, and weighed. The results thus obtained varied from 0·65 to 0·70 gramme. The loss, through solubility of the powder in the glycerin, amounted, in the mean of several determinations on 1 gramme of pure lentiscus, to 30 per cent., and on adding this to the amount actually found, the results agreed well with the quantity taken.

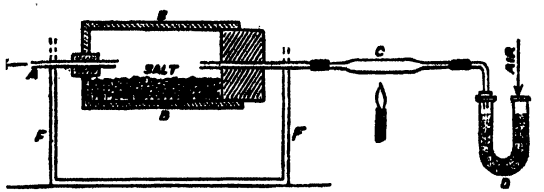
It was not found possible to quantitatively estimate tamarix leaves in this way, but they could be identified. For this purpose 5 grammes of the sieved sample were introduced into the tube of glycerin, which was then boiled as described above, and allowed to stand for two hours. If the supernatant glycerin was then clear and of a yellowish-green colour, the sumach was regarded as pure. If, however, after twenty-four hours part of the powder remained on the surface, while the glycerin was clear and of a wine-red colour, the presence of lentiscus leaves was, in the author's experience, certain. Finally, when tamarix leaves were the adulterant, part of the powder remained after two hours in suspension in the glycerin, which had the same wine-red colour as in the case of lentiscus. After twenty-four hours the suspended powder completely subsided. C. A. M.

INORGANIC ANALYSIS.

Direct Determination of Carbon in Metallic Alloys. H. Brearley. (*Chem. News*, vol. lxxxi., pp. 91, 92.)—Samples of various alloys were heated for one hour in a Bunsen furnace, the porcelain tubes being supported in asbestos arches, thus enabling a higher temperature to be maintained than usual with combustion tubes. The results showed the method to be applicable to metallic tungsten, ferro-tungsten, ferro-silicon, 12 per cent. silicon-spiegel, ferro-silico-molybdenum, and 20 per cent. spiegel, though the last-named could not be completely oxidized. On the other hand, metallic molybdenum volatilizes, leaving only impurities like SiO_2, WO_3, etc.; tungsten-nickel alloy is imperfectly oxidized, and ferro-manganese, ferro-chromium, chromium nickel and metallic chromium cannot be oxidized without some other oxidizing reagent. Ferromanganese is oxidized completely in presence of oxides inert towards chromium alloys, *e.g.*, zinc oxide. C. S.

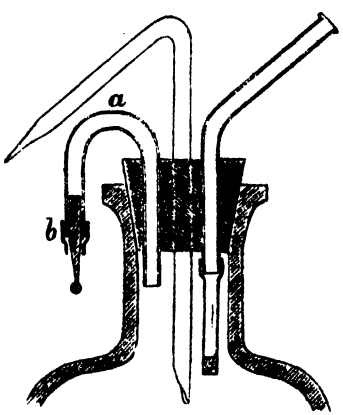
APPARATUS.

New Device for drying Crystallized Salts. F. H. Getman. (*Chem. News.*, vol. lxxxi., p. 91.)—The apparatus consists of a large porous cup, B B, to contain the salt to be dried, with a hole in the bottom to take a good-sized cork, the mouth being also closed with a cork. Glass tubing is fitted centrally through both corks, and, the whole being placed in a frame or box F F, the tube A is connected with an aspirator, whilst the other tube is connected with the drying tube D, with or without the intermediate tube C, which can be heated to any desired temperature when hot air is required. The cup may be slowly rotated from time to time to expose a fresh surface of the salt to the air current.



C. S.

An Improved Wash-Bottle with Bunsen Valve. O. Gamber. (*Chem. Zeit.*, 1900, xxiv., 395.)—When an ordinary wash-bottle is fitted with a Bunsen valve, it is impossible to arrest the stream of water suddenly without loosening the cork of the flask, unless the exit tube is provided with a length of rubber which can be pinched with a clip or with the fingers. The annexed sketch represents an improvement that brings the current of liquid under more immediate control. *a* is a bent piece of glass tube, carrying at its outside end a short length of rubber, *b*, the normal diameter of which is about one-half that of the glass. Within the glass tube slides easily a conical piece of rod (shaded in the diagram), which has a maximum diameter less than that of the glass, but greater than that of the rubber. When there is an excess of pressure in the wash-bottle, the rod is forced outwards till it makes a joint against the rubber tube; but if the finger give a slight lifting movement to the ball at the end, the rod slips upwards, the joint is broken, the pressure relieved, and the stream of water stops.



F. H. L.

Aluminium Plates for Laboratory Purposes. Fritsch and Venator. (*Chem. Zeit.*, 1900, xxiv., 286.)—The authors recommend sheets of aluminium 3 or 4 millimetres thick as substitutes for the ordinary wire gauze or sand bath. They have employed such a plate for five years in a fume cupboard, and it is still in perfect condition. The efficiency of the gas-burner is much greater than with a sand bath, while dust and breakage of the gauze are avoided. When evaporating liquids to dryness, a thin sheet of asbestos should be laid over the metal.

F. H. L.

INSTITUTE OF CHEMISTRY OF GREAT BRITAIN AND IRELAND.

THE following is a list of the names of the candidates who passed the examination of the Institute of Chemistry held in July, 1900:

INTERMEDIATE EXAMINATION.—Brown, J. A., University College, Nottingham. Chapman, E. M., Pharmaceutical Society's Laboratories, and King's College, London. Cunliffe, F., Owens College, Manchester. Dewhirst, J. A., Yorkshire College, Leeds, and Pharmaceutical Society's Laboratories. Dick, W. D., King's College, London. Ellis, A. W., Mason University College, Birmingham. Gray, A., Glasgow and West of Scotland Technical College. Jenkins, J. E., King's College, London. Lowson, W., Yorkshire College, Leeds. Lumsden, W. W., Glasgow and West of Scotland Technical College. McLellan, B. G., Glasgow and West of Scotland Technical College. Nuttall, W. H., University College, Nottingham. Partridge, W., Finsbury Technical College, London. Richmond, S. O., Finsbury Technical College, London. Robertson, A. J., University College, Dundee, and under R. R. Tatlock, Esq., F.I.C. Tainsh, P. W., Glasgow and West of Scotland Technical College. Tebb, W. S., M.A., M.D. (Cantab.), Cambridge University and King's College, London. Thorburn, J., Glasgow and West of Scotland Technical College. Watson, F. W., Glasgow and West of Scotland Technical College.

GENERAL PRACTICAL EXAMINATION.—Scott, A.,* Glasgow University, Glasgow and West of Scotland Technical College, and under R. R. Tatlock, Esq., F.I.C.

FINAL A.I.C. EXAMINATION.—*In Mineral Chemistry:* Baguley, A., B.Sc. (Wales), University College, Bangor. Davidson, A., Glasgow and West of Scotland Technical College. Hartley, T.,* Yorkshire College, Leeds. Kinnersley, H. W., Merchant Venturers' Technical College, Bristol, King's College, London, and under E. H. Cook, Esq., D.Sc. (Lond.), F.I.C. Reinherz, O., B.A. (Cantab.), Trinity College, Cambridge. *In Metallurgical Chemistry:* Levy, A. G., Finsbury Technical College, London. Morgan, J. J.,* of Wednesbury. *In Physical Chemistry:* Pope, T. H., A.C.G.I., City and Guilds of London Institute, Central Institution, and Finsbury Technical College, London. *In Organic Chemistry:* Billows, F. G. H., A.C.G.I., City and Guilds of London Institute, Central Institution, and Finsbury Technical College, London. *In the Analysis of Food and Drugs, including an Examination in Therapeutics, Pharmacology and Microscopy:* Aston, S., University College, London. Booth, N. P., Mason University College, Birmingham. Glass, T. W.,* B.Sc. (Lond.), Pharmaceutical Society's Laboratories, and under Messrs. T. H. Redwood and A. J. de Hailes, FF.I.C. Jollyman, W. H., Finsbury Technical College. Platt, W. N.,* B.Sc. (Lond.), A.R.C.Sc. (Lond.), Royal College of Science, London. Woodhead, S. A.,* B.Sc. (Dun.), Durham College of Science, Newcastle-on-Tyne.

The examiners in chemistry were Dr. Bernard Dyer, D.Sc. (Lond.), F.I.C., and Dr. W. Palmer Wynne, D.Sc. (Lond.), F.R.S., F.I.C. The examiner in therapeutics, pharmacology and microscopy was Dr. Thomas Stevenson, M.D. (Lond.), F.R.C.P., V.-P.I.C.

* For the Fellowship (F.I.C.).

THE ANALYST.

SEPTEMBER, 1900.

PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS.

THE COMPOSITION OF MILK AND MILK-PRODUCTS.

By H. Droop Richmond.

(*Read at the Meeting, May* 2, 1900.)

Of the 34,943 samples analysed in the Aylesbury Dairy Company's Laboratory during 1899, 29,256 were analyses of milk.

The composition of 14,133 samples taken on arrival of the milk from the farms at the chief and branch depôt is given in Table I. The composition of the morning and evening milk is kept separate.

Table I.

Average Composition of Milk during 1899.

Month.	Morning Milk.				Evening Milk.				Average.			
	Specific Gravity.	Total Solids.	Fat.	Solids-not-fat.	Specific Gravity.	Total Solids.	Fat.	Solids-not-fat.	Specific Gravity.	Total Solids.	Fat.	Solids-not-fat.
January	1·0326	12·68	3·68	9·00	1·0324	13·05	4·02	9·03	1·0325	12·86	3·85	9·01
February	1·0326	12·66	3·65	9·01	1·0324	13·05	4·01	9·04	1·0325	12·85	3·83	9·02
March	1·0326	12·57	3·59	8·98	1·0324	12·90	3·90	9·00	1·0325	12·74	3·75	8·99
April	1·0325	12·56	3·58	8·98	1·0324	12·85	3 84	9·01	1·0325	12·70	3·71	8·99
May	1·0328	12·38	3·38	9·00	1·0326	12·80	3·77	9·03	1·0327	12·59	3·57	9·02
June	1·0327	12·26	3·30	8·96	1·0322	12·63	3·70	8·93	1·0325	12·45	3·50	8·95
July	1·0320	12·30	3·47	8·83	1·0315	12·51	3·75	8·76	1·0318	12·40	3·61	8·79
August	1·0315	12·32	3·60	8·72	1·0309	12·47	3·83	8·64	1·0312	12·40	3·72	8·68
September	1·0319	12·51	3·67	8·84	1·0315	12·84	4·03	8·81	1·0317	12·68	3·85	8·83
October	1·0325	12·70	3·70	9·00	1·0322	13·04	4·06	8·98	1·0324	12·87	3·88	8·99
November	1·0323	12·64	3·69	8·95	1·0321	12·97	4·01	8·96	1·0322	12·80	3·85	8·95
December	1·0326	12·56	3·59	8·97	1·0324	12 91	3·90	9·01	1·0325	12·74	3·75	8·99
Average	1·0324	12·51	3·57	8·94	1·0321	12·83	3·90	8·93	1·0322	12·67	3·74	8·93

As has been noticed in former years, the lowest fat occurs in June and the highest in October. During the months of July and August the solids-not-fat are

below the average, as has been recorded in every previous year. Last year, owing to the drought, the depression in solids-not-fat extended into September, while this year the September solids-not-fat show a marked increase over those of August. As the September of 1899 was a comparatively wet month, this is further evidence that drought has a tendency to cause low solids-not-fat.

Three samples representative of the mixed bulk of milk from a whole farm fell below 3·0 per cent. of fat, the lowest figure recorded being 2·94, and many samples, especially during July and August, fell below 8·5 per cent. solids-not-fat.

Three samples especially may be mentioned. Of these the first is from a single cow with a suspicious udder, whose calf died and was found to be in an advanced stage of tuberculosis; this sample was not obtained from the herds supplying the company, but was obtained from a private source. The other two are from milk taken under the personal supervision of the lady to whom the herd belonged.

	I.	II.	III.
Specific gravity	1·0302	1·0288	1·0290
Total solids	10·66	11·39	11·50
Fat	2·60	3·37	3·40
Sugar	3·23	4·03	4·08
Proteids	3·98	3·24	3·27
Ash	0·85	0·75	0·75
Solids-not-fat	8·06	8·02	8·10

These samples confirm the view previously expressed, that when genuine samples are low in solids-not-fat, the proteids and ash are normal, and the milk-sugar is the constituent on which the deficiency falls.

I have in a previous paper (ANALYST, xxiii., 169) drawn attention to the fact that the expression G + F is approximately a constant; this does not hold in abnormal samples. I would here mention that all milks, abnormal or otherwise, conform to the following two rules, which are practically different modes of expression of the same fact.

A. The solids-not-fat, less the milk-sugar, calculated on the milk devoid of fat, amount to at least 4·0 per cent. This, expressed as a formula, is:

$$(S - L)\frac{100}{100 - F} \text{ exceeds } 4{\cdot}0.$$

(S = solids-not-fat, L = milk-sugar, F = fat.)

B. The sum of the specific gravity degrees and the fat, less four times the milk-sugar, exceeds 16·0; this, expressed as a formula, is:

$$G + F - 4L \text{ exceeds } 16{\cdot}0.$$

The following Table II. gives analyses which support this view. The bulk of the analyses have been previously published, and the references are given for verification.

TABLE II.

Degrees of Specific Gravity. G.	Fat. F.	Sugar. L.	Solids-not-fat. S.	(A) $S - L \times \frac{100}{100 - F}$	(B). $G + F - 4L$.	Reference.
33·0	3·30	4·77	8·85	4·21	17·22	ANALYST, xxiv., 198.
32·0	3·60	4·70	8·71	4·13	16·80	,, ,,
32·5	3·35	4·70	9·06	4·50	17·05	,, ,,
32·5	3·85	4·82	8·88	4·21	17·07	,, ,,
32·3	3·05	4·81	8·74	4·05	16·11	,, ,,
33·0	3·30	4·84	8·94	4·14	16·94	,, ,,
33·2	3·15	4·92	9·13	4·34	16·67	,, ,,
32·6	3·85	4·63	9·00	4·53	18·03	,, ,,
32·6	2·95	4·58	8·69	4·23	17·23	,, ,,
32·7	3·35	4·75	8 77	4·15	17·05	,, ,,
32·8	3·95	4·82	8 85	4·19	17·47	,, ,,
34·2	3·50	4·86	9·14	4·53	18·26	,, ,,
33·0	3·40	4·66	8·98	4·47	17·76	,, ,,
32·0	3·53	4·66	8·83	4·32	16·89	,, ,,
32·9	3·70	4·60	9·09	4·66	18·20	,, ,,
32·5	3·25	4·68	8·85	4·31	17·03	,, ,,
32·7	3·35	4·88	8·91	4·16	16·57	,, ,,
31·5	3·75	4·56	8·85	4·45	17·01	,, ,,
31·5	3·15	4·50	8·71	4·34	16·65	,, ,,
32·0	3·35	4·58	8·63	4·18	17·03	,, ,,
31·8	3·65	4·63	8·78	4·30	16·93	,, ,,
32·2	3·40	4·65	8·78	4·27	17·00	,, ,,
32·0	3·65	4·85	8·96	4·26	16·25	,, ,,
32·5	3·65	4·75	9·14	4·55	17·15	,, ,,
33·5	3·55	4·87	8·84	4·11	17·57	,, ,,
32·5	4·25	4·74	8·93	4·37	17·79	,, ,,
32·7	3·55	4·72	8·80	4·22	17·37	,, ,,
32·3	3·90	4·79	8 90	4·27	17·04	,, ,,
32·0	4·00	4·85	8·83	4·14	16·60	,, ,,
32·2	4·55	4·50	8·87	4·52	18·75	,, ,,
32·0	4·15	4·70	8·91	4·39	17·35	,, ,,
31·6	5·35	4·54	8·69	4·38	18·81	,, ,,
32·2	3·55	4·66	8·70	4·18	17·11	,, ,,
31·5	4·00	4·54	8·72	4·37	17·34	,, ,,
31·3	3·55	4·54	8·65	4 25	16·69	,, ,,
31·3	4·00	4·65	8·73	4·24	16·50	,, ,,
32·3	4·10	4·78	8·88	4·26	17·28	,, ,,
32·6	3·60	4·76	8·93	4·35	17·16	,, ,,
32·2	3·75	4·66	8·78	4·28	17·31	,, ,,
32·0	3·65	4·56	8·58	4·17	17·41	,, ,,
32·5	3·50	4·96	8·89	4·07	16·16	,, ,,
32·0	3·90	4·67	8·75	4·24	17·22	,, ,,
32·3	3·60	4·72	8·87	4·30	17·02	,, ,,
31·5	3·85	4·64	8·72	4·24	16 77	Not published.
28·2	6·77	4·48	8·35	4·16	17·05	,, ,,
27·1	8·37	4·31	8·43	4·50	18·17	,, ,,
34·3	4·27	4·79	9·28	4·69	19·41	,, ,,
32·4	7·65	4·94	9·10	4·51	20·29	,, ,,
29·5	5·69	4·14	8·25	4·36	18·63	,, ,,
30·8	3·99	4·41	8·53	4·29	17·15	,, ,,

Degrees of Gravity. G.	Fat. F.	Sugar. L.	Solids-not-fat. S.	(A) $S - L \times \frac{100}{100 - F}$	(B) G + F − 4L.	Reference.
33·6	3·08	4·89	9·12	4·36	17·12	Not published.
33·6	3·77	5·17	9·16	4·15	16·69	,, ,,
33·5	3·71	4·66	9·08	4·59	18·57	,, ,,
			Abnormal Milks.			
30·7	4·00	4·48	8·44	4·12	16·78	ANALYST, xxiv., 198.
31·8	3·20	4·77	8·55	3·90	15·92	,, xxiv., 199.
28·5	4·04	3·65	7·99	4·63	17·94	,, ,,
29·8	4·14	3·89	8·39	4·69	18·38	,, ,,
28·8	4·39	3·82	8·17	4·54	17·91	,, ,,
29·5	3·94	3·84	8·29	4·63	18·08	,, ,,
28·2	4·81	3·70	8·09	4·60	18·21	,, ,,
36·3	3·62	4·61	10·24	5·79	21·28	(Vieth) ANALYST, xvii., 89 (Bodmer)
24·8	3·14	2·59	7·24	4·80	17·58	ANALYST, xx., 266.
30·2	2·60	3·23	8·06	4·96	19·88	See *ante*.
28·8	3·37	4·03	8·02	4·13	16·05	,,
29·0	3·40	4·08	8·10	4·16	16·08	,,

H. Timpe (*Chem. Zeit.*, 1899, 1040) has lately drawn attention to the fact that there is a constant relation between the proteids and fat in genuine milk, expressed by the formula $P = 2 + 0{\cdot}35\ F$; and he proposes to detect adulteration in milk by a divergence between the proteids found and calculated.

If the difference (found *minus* calculated) is positive, and exceeds in mixed milk +0·06, he concludes skimming; if negative, and exceeding −0·06, he concludes dilution either by excess of fat or by water, and decides between these two causes by the sugar and ash determinations.

He deduces his formula from twenty-one analyses of "normal" milks, in which the differences vary from +0·12 to −0·11. He gives analyses in his paper of forty abnormal milks, the first nine of which are classed as abnormal because the cows were either stall fed or experimentally fed, the next fifteen samples because the cow was diseased (and these are rightly classed as abnormal); the last sixteen samples were derived from a cow which had recovered from gastric catarrh.

He also quotes feeding experiments by Langbeck on three cows: one cow remained of the same weight, and gave milk of fairly constant composition throughout the series, and he points out that the milk of this cow agreed with his formula except when the food was changed; the other two cows altered in body-weight, and their milk constantly disagreed with his formula, and was therefore classed as abnormal.

He concludes that normal milk—*i.e.*, milk produced from grass-fed cows, whose food is not changed, and who are not suffering from disease—will, when the mixed product of several cows is considered, agree with his formula within ±0·06 per cent. (though one-third of his normal milks exceed this). This conclusion involves the assumptions that stall-feeding, change of ration, etc., are practically unknown, and that the methods of analysis involve an experimental error of less than 0·06 per cent. These assumptions appear to be unwarranted. Certainly in English dairy practice

stall-feeding and change of diet are largely practised; and if we admit his results with stall- and experimentally-fed cows as "normal"—and there appears no reason to suppose them otherwise—the percentage of disagreement with his formula jumps up to over 50.

If we take the results obtained in the estimation of the fat and proteids, an experimental error of 0·1 per cent. is by no means an over-estimate of possible variations; an error of 0·1 per cent. in each might mean a variation of 0·135 per cent. in the difference, a figure which is more than double his allowable variation.

When his series is extended—and I have done this by applying his formula to over fifty milks—the agreement practically disappears. The following table expresses my results in a condensed form:

Percentage of Fat.	Percentage of Proteids. Extremes.	Mean.	Calculated.	Difference.
3·0 -3·25	3·11–3·48	3·35	3·08	+0·27
3·25–3·50	3·22–3·60	3·38	3·19	+0·19
3·50–3·75	3·27–3·76	3·46	3·27	+0·19
3·75–4·00	3·30–3·63	3·40	3·36	+0·04
4·00–4·25	3·27–3·68	3·49	3·44	+0·05
4·25–4·50	3·45–3·55	3·50	3·60	−0·10
Above 4·50	3·42–3·66	3·52	3·77	−0·25

The maximum individual differences varied from +0·46 to −0·44, or nearly 0·5 per cent.

It is seen from the above table that there is a very slight tendency for the proteids to be higher when the fat is high, but the tendency is very much less than that indicated by Timpe's formula.

In my opinion, the conclusions arrived at in the paper are entirely unwarranted by the facts, because—

(*a*) The number of samples of "normal" milk is too small to admit of a general conclusion being drawn.

(*b*) The limitations within which the formula is applicable are so narrow as to destroy its usefulness in commercial work, and the "abnormal" samples are classed as such on insufficient grounds.

(*c*) The agreement expected is greater than the accuracy with which commercial analysis can be done.

(*d*) On applying the formula to results obtained under other conditions it fails utterly.

The bulk of the fat estimations of the milks referred to in Table I. were made by the Gerber method; though the accuracy of this is generally admitted, I have thought that some figures, showing the differences between this and gravimetric methods during 1899 would show the extent to which the figures are reliable.

In all 65 experiments where comparisons were made are available; the fat varied from 1·98 per cent. to 4·60 per cent. The maximum differences were +0·15 and −0·20, with a mean difference of −0·016.

Of the 65 samples 39 had an error of 0·05 or less = 60·0 per cent.; 15 an error of 0·06 to 0·10 = 23·1 per cent.; 9 an error of 0·11 to 0·15 = 13·9 per cent.; and 2 an error of 0·16 to 0·20 = 3·1 per cent.

The two samples with differences of more than 0·15 were a human milk and a somewhat clotted sterilized milk, and excluding these, and including only the 68 ordinary milks, the maximum differences were +0·15 and −0·14, and the mean was −0·01 per cent.

Storch holds that the fat globules in milk are surrounded by a "mucoid" membrane; I have already expressed myself as unable to accept this view (ANALYST, xxii., 207). If Storch's view were correct, it would be expected that the membrane would bear such a proportion to the smallest fat globules that their density would be equal to that of the milk serum, and the last traces of fat could not be removed by centrifugal force. By means of the Crown Hand Separator, which is very efficient, I have succeeded, on running milk twice, in obtaining samples of separated milk in which the percentage of fat is so small that it does not reach the second place of decimals per cent.; this fact, while not definitely disproving Storch's view, is further evidence against it.

A sample of separated milk was run for fifteen hours in a centrifugal machine at the rate of 1,000 revolutions per minute. Cultivations on gelatine were made from the top portion, the middle, and the bottom. The results were, after eight days of at 22°:

	Colonies per $\frac{1}{1000}$ c.c.	Remarks.
Top	197 ...	Growth rapid, about 20 per cent. liquefied.
Middle ...	5	
Bottom ...	194 ...	Growth slow, none liquefied.

This appears to show that while some micro-organisms have a density greater than 1·036, others have a less density. The top cultivation was made from the portion immediately underneath the thin layer of cream, so that it is not probable that they were carried up by the cream.

The composition of clotted cream during 1899 was found to be:

Constituent.	Maximum.	Minimum.	Average.
Total solids	71·58 ...	60·75 ...	65·74
Fat	64·70 ...	50·27 ...	58·24
Ash	0·83 ...	0·46 ...	0·60
Solids-not-fat	10·48 ...	5·67 ...	7·50

The composition agrees with that found in previous years.

The following percentages of water in butter have been found:

	Maximum.	Minimum.	Average.
French, fresh	15·10 ...	13·08 ...	14·32
Irish, fresh	14·09 ...	11·79 ...	12·87
Irish, salt	13·29 ...	11·33 ...	12·31
Danish, salt	13·17 ...	10·63 ...	11·93

Two samples of butter prepared by a "butter extractor" which was claimed to increase the yield of butter was examined. The analyses were:

	I.	II.
Fat	77·86 ...	—
Water	20·42 ...	23·16
Curd	1·49 ...	—
Salt	0·23 ...	—

It is not difficult to see how the increased yield is obtained.

The following average figures were determined on the fat with a Zeiss butyro-refractometer:

	Refraction Number at 35°.	KOH absorption.
French butter	46·4°	22·88 per cent.
Irish ,, ...	46·4°	
Danish ,, ...	46·2°	

A sample of butter prepared from the milk found last year to be low in solids-not-fat was found to have a refraction number of 47·2° at 35°, and a KOH absorption of 22·12 per cent.

Some experiments were carried out, chiefly by Mr. C. H. Rosier, on the reaction described by Dupouy (ANALYST, xxii., 211) with paraphenylene-diamine for distinguishing between fresh and boiled milk; none of the usual preservatives added to milk were found to interfere with this reaction, though in the presence of formaldehyde the colour had a somewhat grayish tint. It was found that metaphenylene-diamine also gave a blue colour less strongly developed than when the para-compound was used. On adding amyl alcohol, shaking, and allowing to separate, the colour was in each case taken up by the amyl alcohol, but while the colour turned brown with the para-compound, it remained blue with the meta-compound. Alkalies turned the colours red. We found that the amyl alcohol solution of the colour from the para-compound turned brown very rapidly when exposed in thin layers to the air, while the solution with the meta-compound remained blue, and the amyl alcohol could be evaporated without much change. Oxidizing agents, such as potassium permanganate, produced a blue compound with both para- and meta-phenylene-diamine.

It appears probable than an oxydase which is destroyed by heat, exists in milk and this produces an unstable blue compound with para-phenylene-diamine, and a stable one with meta-phenylene-diamine; the addition of amyl alcohol increases the delicacy of the test with the latter; we recommend meta-phenylene-diamine, together with amyl alcohol, as the best reagent.

A sample of separated milk was met with which did not give this reaction, either with para- or meta-phenylene-diamine, although it had not been heated above the temperature necessary for separation; it also curdled with difficulty with rennet, and contained abundance of soluble albumin. The colour tests for heated milk should not be implicitly relied upon, and Faber's albumin test (ANALYST, xiv., 141) should always be applied before concluding that milk has been heated.

Before concluding I would mention that the bulk of the work has been performed by Messrs. F. R. O'Shaughnessy, J. B. P. Harrison, and C. H. Rosier.

DISCUSSION.

The PRESIDENT having invited discussion,

Dr. DYER desired to ask if the author knew what was the breed of the cows that yielded the samples of abnormal milk referred to. He (Dr. Dyer) was inclined to think that the use of some of the formulæ mentioned was in the direction of unnecessary elaboration. In the case of the first formula, for instance, it seemed much simpler to say that, in the fat-free milk, the percentage of proteids and ash was usually above 4, which seemed really to be all that the formula conveyed. Criticism

of this kind, however, did not detract from the appreciation to which the work involved in this series of papers was entitled.

Mr. T. H. POPE desired to point out that in the second formula the limits given for the abnormal milk were inside those in the normal milk, whereas in the case of the first formula they were very close. He understood the author to say that these formulæ would be of use in doubtful cases; but, having regard to these numbers, he hardly thought that that would hold.

Mr. JOHN WHITE said that he too had found that butter containing an excessive quantity of water—*i.e.*, over 16 per cent.—was becoming of less frequent occurrence than formerly. Out of a large number of samples of butter of colonial, Irish, Danish, German and French origin, which had been submitted to him under the Sale of Food and Drugs Act within the last two years, he had only met with two in which the proportion of water was as much as 17 per cent. He also had examined samples of butter made by a process which was supposed not only to yield more butter than ordinary processes, but to yield it in a much shorter time; and one of those samples contained 28 per cent. of water, being in this respect somewhat similar to the samples referred to in the paper.

The PRESIDENT (Mr. FISHER) said that, having had occasion to refer to the former papers of this series for the purposes of the evidence which he had recently laid before the Milk Standards Committee, he felt personally indebted to the author for the large quantity of information which they contained. In reference to the formula $p = 2 + 0{\cdot}35\ f$, it seemed that while, in the case of milk containing about 4 per cent. of fat, the average percentage of proteids was indicated fairly correctly, a larger percentage would be indicated as the fat increased, and a smaller percentage as the fat diminished. But this would not afford any clue to the original composition of a sample of milk from which, for instance, a portion of the fat had been artificially removed. The difference between morning and evening milk was a point of great practical importance as affecting the question of standards and limits, having regard to the small proportions of fat sometimes found in milks which were mixed milks, and yet which must be accepted as genuine.

Mr. RICHMOND, in reply, said that he had no information as to the breed of the cows which yielded samples No. 2 and No. 3. No. 1 was the milk of a Jersey cow. The formula referred to by Dr. Dyer had been introduced because the figure calculated from it might to a certain extent be substituted for the percentage of proteids obtained by direct determination. In the case of formula No. 2, any experimental error in the milk-sugar estimation was multiplied four times, whereas in formula No. 1 it was not multiplied at all, which accounted for the fact that the two formulæ did not give strictly comparative results. Furthermore, any variation in the percentage of fat would affect formula No. 2 and not No. 1, seeing that in No. 1 the milk-sugar was expressed in terms of the fat-free milk, while in No. 2 the milk-sugar was taken as it stood. As, however, the constancy of the results of both formulæ was only approximate, he had not thought it necessary to put them in exactly the same form. He would like to make it quite clear that the samples of butter containing 20·4 and 23·2 per cent. of water were obtained by means of a machine in which the operation of churning was practically instantaneous, whole

placed in the machine, and skim milk running out at one side and butter at the other. He agreed with the President's criticism of the formula $p = 2 + 0{\cdot}35\ f$. As the fat increased, however, there was a slight tendency for the proteids to increase also, though not to anything like so great an extent as the formula represented.

He felt that the fact should be referred to that this series of papers had been commenced by Dr. Vieth, to whom was due in the first instance the system that was now being followed.

ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

FOODS AND DRUGS ANALYSIS.

Estimation of Total Extract in Wine. **A. Hubert.** (*Rev. gén. Chim. pure et appliq.*, 1900, ii., 374; through *Chem. Zeit. Rep.*, 1900, 168.)—The author obtains constant results by absorbing 5 c.c. of the sample in porous filter-paper, and keeping it for four or five hours over sulphuric acid under diminished pressure at a temperature of 50° C. He has devised a convenient arrangement evacuated by means of a water-pump, fitted with thermostat and pressure-gauge, and capable of being placed as a whole inside a vessel full of warm water. Aqueous glycerol can also be evaporated in a similar manner with moderate accuracy. F. H. L.

Vanillin in Wine Vinegar. **A. Stocky.** (*Zeit. für Untersuch. der Nahr. und Genussmittel*, 1900, iii., 235.)—The presence of vanillin being suspected in a sample of wine vinegar, the following procedure was adopted for its detection: A large quantity was evaporated with calcium carbonate, the residue extracted with ether, and the extract evaporated to dryness on the water-bath. The residue was repeatedly re-crystallized from alcohol and identified by the following tests:

(*a*) The production of a metallic mirror with ammoniacal silver solution.

(*b*) The production of phloroglucinvanillin by the addition of phloroglucinol to a moderately strong hydrochloric acid solution of the crystals. H. H. B. S.

Influence of Saccharin upon Peptic and Pancreatic Digestion. **F. Berlioz.** (*Chem. Zeit.*, 1900, xxiv., 416.)—The author's experiments confirm the statement of Nencki (*Farmazeft*, 1899, vii., 1130) that saccharin, at least in small amounts, does not interfere with gastric or pancreatic digestion. F. H. L.

TOXICOLOGICAL ANALYSIS.

Notes on the Detection of Conine in Cases of Poisoning. **D. Vitali and C Stroppa.** (*Boll. Chim. Farm.*, 1900, xxix., 221; through *Chem. Zeit. Rep.*, 1900, 170.)—The authors have recently been engaged in a legal case of the above description, and record the following observations: A one per cent. solution of conine acetate

or tartrate can be distilled without any alkaloid passing over with the When the Stas-Otto method of working is adopted, ether dissolves an appreciable quantity of conine before the liquid is made alkaline, *i.e.*, dissolves alkaloid from an acid solution. Conine can be separated from any colouring matter or ptomaines which may be present, by shaking the ethereal extracts (either from the acid or the alkaline liquors) with water faintly acidified with acetic acid, then making the liquid alkaline with barium hydroxide, and extracting with petroleum spirit boiling between 40° and 60° C. To avoid as much as possible the discoloration of the conine salt on evaporation, it should be treated with lead acetate and sulphuretted hydrogen; the lead sulphide carries down the colouring matter. The characteristic smell of mice is so distinct that it is noticeable on warming a 1 : 100,000 solution. When a few drops of the green solution prepared by dissolving 1 gramme of potassium permanganate in 200 c.c. of strong sulphuric acid are added to a little conine, the colour changes to a violet, and is unaffected by further additions of strong sulphuric acid. Conine gives a white precipitate with Nessler's reagent. With trichloracetic acid in fairly strong solution, it yields a turbidity soluble in excess of acid; on evaporation at a moderate temperature, a residue of microscopic needles (in bunches) is left. Guinea-pigs and frogs die half an hour after the injection of a small dose, exhibiting paralysis, which appears first at the hind quarters. As Rossbach has stated, one drop of a very weak solution of conine is rapidly fatal to the infusoria that develop in water in which oats have been soaked for a few days. F. H. L.

ORGANIC ANALYSIS.

Oil of Mandarin. **G. Fabris.** (*Annali del Laboratorio delle Gabelle*, 1900, iv., 140, 141.)—This oil is rarely obtained pure in commerce, owing to its production being limited and its price high. It is obtained from the peel by hand in the same manner as oil of orange or oil of lemon.

It is a golden-yellow liquid, with a slight bluish fluorescence, which becomes more pronounced on diluting the oil with alcohol. Its specific gravity varies from 0·854 to 0·858 at 15° C., and its rotatory power at 20° C. from +65° to 75°. It begins to boil at 175° C., and distils over almost completely at 179° C. The fraction distilling between 175° and 177° C. has a rotation of 76° to 77°. It consists for the most part of limonene, and also contains small quantities of aldehydes (citral and citronellal).

C. A. M.

Oil of Sweet Orange **G. Fabris.** (*Annali del Laboratorio delle Gabelle*, 1900, iv., 136-139.)—This oil, commonly known as *Essenza di Portogallo*, is principally extracted in Calabria, and in the provinces of Messina, Catania and Syracusa, in Sicily, the peel being squeezed by hand, and the expressed juice caught in a sponge.

It has a more or less pronounced orange colour and an aromatic taste. It consists for the most part (about 90 per cent.) of limonene, and contains citral and possibly another aldehyde, but no pinene. The most characteristic properties of the oil are its specific gravity, rotatory power, and boiling point, which, from the observa-

tion of different authorities, vary within the following limits: Specific gravity at 15° C., 0·848 to 0·852; rotation at 20° C., +96° to +98°; boiling point, 175° to 180° C. The distillate is colourless, and has a slightly lower specific gravity and a slightly higher rotation than the original oil.

Owing to the fact that oil of orange is much more constant in its physical properties than oil of lemon or oil of bergamot, adulteration is more easily detected. Oil of turpentine and oil of lemon, which are the principal adulterants, raise the specific gravity and lower the rotation. In doubtful cases a fractional distillation is useful, since in the case of a pure oil the first distillate has a slightly higher rotatory power than the original oil, whereas in the case of mixtures the rotatory power of the distillate is lower than that of the original mixed oils. C. A. M.

The Aldehydic and Ketonic Constituents of Verbena Oil: "Verbenon." M. Kerschbaum. (*Berichte*, 1900, xxxiii., 885-893.)

I. *Verbena Oil extracted from Chiris Grass.*—When distilled in a current of steam, this is a dark-coloured oil with an odour resembling that of lemon-grass oil. Its specific gravity at 17° C. is 0·903, and its specific rotation = $(\alpha)_D - 12° 30'$. The author found a specimen to contain 26 per cent. of citral and 74 per cent. of terpenes and alcohols. The citral, isolated by Tiemann's method (*Berichte*, xxxi., 3336), contained, in addition to citral *a*, from 17 to 20 per cent. of citral *b*.

II. *Verbena Oil distilled from Spanish plants.*—This oil, which differs somewhat in odour from the preceding oil, has a specific gravity of 0·926 at 17° C., and $(\alpha)_D + 2° 45'$. A specimen examined by the author was found to contain 13 per cent. of citral, 86 per cent. of alcohols and terpenes, and about 1 per cent. of a new optically active ketone to which the name of "verbenon" was given.

This new ketone was obtained as a semicarbazone ($C_{11}H_{17}N_3O$ or $C_{11}H_{19}N_3O$) from the verbena oil, after complete removal of the citral. The semicarbazone (melting point 208-209° C.) yielded, on treatment with phthalic anhydride and steam, "verbenon" as a colourless oil. It had a characteristic odour recalling that of camphor and of peppermint. It was completely insoluble in water, but miscible in all proportions with the ordinary organic solvents.

On oxidation with potassium permanganate it yielded a ketonic acid ($C_9H_{14}O_3$), from which Baeyer's norpic acid, $C_8H_{12}O_4$ (*Berichte*, xxix., 1911) was obtained by treating it with sodium hydroxide and bromine.

It was not definitely determined whether verbenon was a saturated or unsaturated substance, or whether its formula was $C_{10}H_{16}O$ or $C_{10}H_{14}O$. C. A. M.

Examination of Rubber Goods. O. Chéneau. (*Zeits. Unters. Nahrungs. u. Genussm.*, 1900, iii., 312.)

Preliminary Tests.—The homogeneity of any specimen of rubber may be conveniently judged by determining the specific gravity and the ash of various small portions of the sample. As the density of caoutchouc lies between 0·919 and 0·956, a higher gravity points to the presence of mineral matter. The ash should be deter-

mined at the lowest possible temperature; if it be high in amount, a complete analysis is advisable. Some of the material should then be gently ignited and the product examined qualitatively; while in the original substance the mineral matter soluble in acetic acid, the carbon dioxide, and the mercury should be estimated separately, as described below. A sample should be extracted with hot or cold alcohol; if it loses more than 2 per cent. by weight, it may be considered to contain foreign organic matter, or to be of inferior quality. The total sulphur should be determined by Henriques' process (ANALYST, 1899, xxiv., 297), but if the rubber yields only a little ash, Mahler's bomb may be employed to save time. About 50 c.c. of water are placed in the bomb, 1 or 2 grammes of rubber in the crucible; oxygen is admitted to a pressure of 5 or 10 atmospheres, and the material is ignited electrically. The water is then collected, the bomb rinsed out, and the sulphuric acid precipitated with barium chloride.

Systematic Analysis.—(1) A weighed quantity of rubber is treated with warm dilute acetic acid, to which some sugar has been added in order to dissolve red lead. The liquid is decanted through a tared filter, the insoluble matter boiled two or three times with water, brought on to the paper, washed till neutral, dried at 105° C., and weighed. In the filtrate calcium, lead, magnesium, zinc and alkalis may be determined. The proportion of substances really soluble in acetic acid which escape dissolution in this process is so small as to be negligible. If the sample was coloured with ultramarine, part of the sulphur thereof is evolved as sulphuretted hydrogen, part remains suspended in the liquid and passes through the filter. If the odour of H_2S is noticed, the amount of sulphur in a portion of the residue should be determined; deducting this from the total sulphur, the sulphur of the ultramarine and that combined with the substances decomposed by the acetic acid is given. (2) The residue from the acetic acid is boiled for two or three hours with normal alcoholic soda under an inverted condenser, driving off the spirit, adding water, and passing the liquid through a tared filter-paper; this operation is repeated, the insoluble matter is washed with boiling water and with water acidulated with acetic acid, then brought on to the filter, dried at 105° and weighed. The loss in weight represents the rubber substitutes, the sulphur combined therewith, the free sulphur, the natural resins the caoutchouc, added resins, pitch, etc. (3) The residue from the caustic soda is extracted with acetone as long as the solvent is coloured; the acetone is distilled off, the solid dissolved in ether, filtered, evaporated, dried at 100° and weighed. This process yields the unsaponifiable oils (mineral and rosin) and part of any asphaltum in the sample. (4) The residue from the acetone is treated with cold nitrobenzene (30 c.c. per 1 gramme), according to Henriques' method of dissolving "asphaltum" (*Chem. Zeit.*, 1894, xviii., 411, ANALYST, xix., 111); all the latter passes into solution, whether it be natural asphaltum or a product derived from tar. In this process unvulcanized rubber loses 1 or 2 per cent., vulcanized rubber on an average 3 per cent., hard rubber somewhat less. (5) The residue from (4) is extracted with petroleum spirit or chloroform, which dissolves paraffin and the unvulcanized rubber that was not removed by the nitrobenzene. In a part of the residue from (5) sulphur is determined; deducting the yield from the difference calculated in (1), the free sulphur and sulphur combined with rubber substitutes and

with asphaltum is given. According to Helm, the asphaltum contains 8 to 11 per cent. of sulphur combined in an organic form, and 0·36 to 0·4 per cent. combined with bases. (6) The rest of the residue from (5), or the original material simply exhausted with acetic acid and sugar, is boiled for an hour, according to Weber's process, under a condenser with 3 c.c. of chloroform and 50 c.c. of nitrobenzene (per 3 grammes of rubber); after cooling, 100 c.c. of ether are added, the insoluble matter is filtered off, washed with petroleum spirit and ether, dried and weighed. If the rubber is hard or heavily loaded, the liquid may filter badly; in this event the operation should be conducted on a number of small portions, decanting the solutions after subsidence, and employing the residues as below. (7) The residue from (6) will contain the mineral substances insoluble in acetic acid, *e.g.*, talc, kaolin, ochre, barium sulphate, stannic sulphide ("mosaic gold"), cinnabar, lead sulphide and sulphate; sometimes also graphite, ground leather, cork, and vegetable fibres. It should be examined microscopically, submitted to a nitrogen estimation, etc., according to circumstances. In another portion sulphur is determined, and by calculation from that obtained in (5), the sulphur combined with the caoutchouc is given.

Carbon dioxide from the carbonates may be determined by treating the sample with dilute acid, passing the vapours through a tube of pumice and copper sulphate, and absorbing the gas in soda-lime. Or the material can be first attacked with hot chloroform and nitrobenzene, and the CO_2 in the product estimated. If the colour of the goods suggests the presence of cinnabar, the mercury should be estimated by a dry process in the cold nitrobenzene residue. F. H. L.

Influence of Dextrose on the Estimation of Urea by means of Hypobromite. L. Garnier and L. Michel. (*Journ. Pharm. Chim.*, 1900, xii., 53; through *Chem. Zeit. Rep.*, 1900, 208.)—The authors find that the irregularities in the estimation of urea by means of hypobromite may be explained by the presence of dextrose. Hypobromite acts energetically upon dextrose, evolving so much heat that the volume of nitrogen cannot be read off till after half an hour's standing. It is also possible that carbon dioxide may be liberated, which will mix with the nitrogen unless a large excess of alkali is employed. Moreover, the nitrogen is always below its proper quantity, for hitherto no method of liberating all the gas from urea has been discovered.* The Kopp-Yvon process of decomposing urea is very convenient, and although it does not give absolutely correct results, it is fairly accurate, especially if the analysis of a urine be conducted side by side with that of a specimen of pure urea in 3 per cent. solution. Taking 2 c.c. of urine, and calling the volume of nitrogen liberated V', with 2 c.c. of standard 3 per cent. urea solution yielding V c.c. of nitrogen, the weight of urea (p) in 1 litre of urine is

$$p = 0{\cdot}06 \times \frac{V'}{V} \times \frac{1000}{2} = 30\frac{V'}{V}.$$

F. H. L.

* A. H. Allen has shown that by adding sodium cyanate all the nitrogen is evolved.—EDITOR.

Estimation of Oxalic Acid in Urine. Existence of Oxaluric Acid therein. E. Salkowski. (*Zeits. physiol. Chem.*, 1900, xxix., 437; through *Chem. Zeit. Rep.*, 1900, 208.)—In the usual methods of determining oxalic acid in urine, the presence of phosphoric acid is apt to cause inaccuracies; the author therefore employs an immiscible solvent which extracts all the former with only traces of the latter. Five hundred c.c. of unfiltered normal human urine are concentrated to one-third the bulk, cooled, acidified with 20 c.c. of 1·12 hydrochloric acid, and agitated three times with 200 c.c. of a mixture of 9 or 10 volumes of ether with 1 volume of alcohol. The extracts are separated, filtered through a dry paper, and distilled. The residue is treated with 10 or 15 c.c. of water, and cautiously heated on the water-bath in a deep basin until the liquid is clear and resinous matter deposits. The whole is filtered, the insoluble portion washed once or twice with a minimum of water, and the filtrate made faintly alkaline with ammonia; then 1 or 2 c.c. of 10 per cent. calcium chloride solution are added, and finally an excess of acetic acid. After at least twenty-four hours' standing, the calcium oxalate is brought on to an ashless filter-paper, washed, etc., and weighed as CaO. A similar process is available for the examination of fabrics in which oxalic acid is suspected, 250 to 800 grammes being taken for analysis.

When urine is evaporated for a long time, the oxalic acid changes into a form which is no longer capable of extraction by means of ether, but which is reconverted into its original condition on boiling with hydrochloric acid. This substance must be oxaluric acid. Whether the latter actually exists in the urine is still unknown; therefore in estimating oxalic acid, the urine should be acidified during concentration (*ut supra*), and the total yield should be considered to include any oxaluric acid.

F. H. L.

Estimation of Chlorine in the Gastric Juice. G. Meillère. (*Bull. Soc. Chim.*, 1900, [3], xxiii., 404; through *Chem. Zeit. Rep.*, 1900, 176.)—The juice is first filtered or treated in a centrifugal machine, in order that the insoluble matter may be available for further examination. In 10 or 20 c.c. of the clear liquid, the acidity is determined by titration with barium hydroxide and phenolphthalein, calculating the result as HCl. To find the total chlorine, 10 grammes of juice are mixed with a small excess of calcium carbonate and 0·2 or 0·25 gramme of calcium nitrate, then evaporated to dryness, and ignited gently till the carbon is oxidized. The residue is taken up with water containing a little acetic acid, neutralized with chalk, and the liquid titrated with silver nitrate and potassium chromate. To estimate the chlorine in the dry residue of the juice, the sample is evaporated *in vacuo* rather than on the water-bath, and treated as above. The chlorine of the ash is determined by evaporating and igniting 10 c.c., next adding calcium nitrate and burning off the carbon, and finally titrating with silver.

F. H. L.

Detection of Salicylic Acid in Presence of Citric Acid. O. Langkopf. (*Pharm. C.H.*, 1900, xli., 335; through *Chem. Zeit. Rep.*, 1900, 176.)—Citric acid or a citrate prevents the ferric chloride reaction for salicylic acid appearing. A fruit

syrup in which the presence of the latter is suspected should therefore be extracted with equal parts of ether and petroleum spirit, and the preservative sought for after the solvents have been driven off. F. H. L.

Detection of Salicylic Acid in Presence of Citric Acid. A. Conrady. (*Apoth. Zeit.*, 1900, xv., 412; through *Chem. Zeit. Rep.*, 1900, 193.)—The failure of the ferric chloride test for salicylic acid when citric or tartaric acid is present, to which Langkopf has referred (see preceding abstract), is presumably due to the formation of some double compound in which the iron exists in the ferrous state. There is no necessity to treat the material under examination with an immiscible solvent; addition of one drop of nitric acid or of hydrogen peroxide suffices to determine the usual reaction. F. H. L.

Relative Value of different Combustibles. F. Isaac. (*Bull de l'Ass. belge*, 1900, xiv., 19-23.)—The general composition of combustibles, after deduction of water and ash, is shown in the subjoined table:

Dry Substance minus Ash.	Carbon.			Hydrogen.	Oxygen, including Nitrogen.	Ratio O/H	Calories Per Kilo.
	Total.	Fixed.	Volatile.				
Wood	50	25	25	6	44	7	5,000
Turf	60	27	33	6	34	6	6,000
Lignite	73	31	42	5·8	21·2	5	7,000
Coal, from the upper measures, dry, burning with much flame	75	60	15	5·5	19·5	4	8,000
Coal, intermediate ...	85	75	8	3 to 5	3 to 20	2 to 3	8,500
Coal, from the lower measures, burning without much flame, anthracitic	95	93	2	2·5	2·5	1	9,000?
Petroleum Oil ...	83	—	83	4	4	0·3	11,000

As regards the samples of compressed lignite which he has examined, the author has met with three different brands, but all having substantially the same appearance, density, and composition—viz., water, 15; ash, 5; volatile matter, 55; and fixed carbon, 25 per cent.; specific gravity, 1·25.

The dry lignite free from ash has the composition shown in the table, and from this the calorific power of the coal per kilogramme can be calculated by the formula of Dulong:

$$\left[8{,}080 \times \frac{\text{H}}{\text{C}} \times 73 + 34{,}500 \times \left(\frac{\text{H}}{5{\cdot}8} - \frac{\text{O}}{8} \right) \frac{21{\cdot}2}{} \right] \div 100 = 6{,}985 \text{ calories, or, in round numbers, } 7{,}000 \text{ calories.}$$

C. A. M.

Estimation of Sulphur in Petroleum by means of Mahler's Bomb. G. Pfeiffer. (Reprint from *Bull. Soc. sciinte din Bucuresci*, 1900, viii., [6]; through

Chem. Zeit. Rep., 1900, 207.)—The above apparatus may conveniently be used to determine the proportion of sulphur in petroleum. In the bomb are placed 2 or not exceeding 2·5 grammes of the material, and oxygen is admitted to a pressure of 30 atmospheres. In the original article Pfeiffer gives a table of the physical and chemical constants of various Roumanian and Russian petroleums, whence it appears that crude Baicoiu oil contains the maximum of sulphur (0·484 per cent.), and the Russian "Wladikawkas" oil the least (0·100 per cent.). The latter, after removal of spirit, is employed for locomotive firing. F. H. L.

Vandyke Brown. H. Borntraeger. (*Oesterr. Chem. Zeit.*, 1900, iii., 294.)—According to the author, Vandyke brown, or Cassel brown (earth), consists essentially of humic acid; and it may therefore be identified, or its purity judged, by its ready solubility in alkali-metal hydroxides or carbonates. It floats on water, is entirely free from sulphur, and leaves a perfectly white ash when ignited with sulphur and potassium nitrate or chlorate. It may be examined by heating 0·5 gramme to 110° C. to determine the moisture, and then boiling it with 2 grammes of caustic potash and 100 c.c. of water for an hour, weighing the undissolved residue. Vandyke brown must not be confused with Cologne earth, which is a manganese colour. F. H. L.

[In his "Chemistry of Paints and Painting," Professor Church describes three kinds of Vandyke brown, none of which corresponds exactly with the above, as all contain more or less iron. Cassel brown is represented by Church to be synonymous with Cologne (Cullen) earth—an impure variety of lignite. Yet "Kölnische Erde" is given by him as the German equivalent of umber.—F. H. L.]

Measurement of the "Fastness" of Printing Inks. (*Oest. Chem. Zeit.*, 1900, iii., 265-267.) — The dry pigment is ground up with one half to one quarter the quantity of linseed-oil varnish, and is printed on to a piece of rag-paper free from wood-fibre. The pigment with which it is to be compared is treated in the same way. The printed surfaces are partly covered with black paper, and are exposed to diffused daylight until the standard colour is almost entirely faded. The intensity of the colour in both the exposed and covered portions is measured by means of a colorimeter. The percentage of the initial colour lost by the standard piece is divided by the percentage lost by the sample. The resulting number the author calls the "fastness" (Lichtechtheit).

If the pigment be very fast as compared with the standard, it is necessary to renew the paper printed with the latter. The percentage loss of colour is measured in each paper, these percentages are added together, and the sum is divided by the percentage loss in the paper printed with the pigment under examination.

NOTE BY ABSTRACTOR.—The author does not state that the comparative "fastness" of one pigment to another is constant when the exposure is varied. It appears improbable that it should be so, especially when the fading is allowed to proceed very far. The numbers obtained by the above method are therefore of doubtful

valve. See Scheurer and Brylinski (*Bull. Soc. Ind. Mulhouse*, 1898, 273) and G. Lemaine (*Ann. Chim. Phys.*, 1895, vi., 433). A. M.

INORGANIC ANALYSIS.

Determination of Slag in Iron and Steel. L. Schneider. (*Oesterr. Zeits. Berg u. Hüttenw.*, 1900, xlviii., 257 and 275; through *Chem. Zeit. Rep.*, 1900, 167.)—Processes which depend on igniting the metal in chlorine or dissolving it in acids yield low results, as they cause a loss of slag; processes which depend on the use of copper or mercuric chloride, etc., only attack portions of the steel very slowly, and give high results. The following is the best method to adopt, as it yields 98 to 99 per cent. of the true slag present in the metal: In a 200 or 300 c.c. beaker immersed in a larger vessel full of cold water is placed 15 c.c. of bromine and 100 c.c. of water; 5 grammes of the sample in turnings are introduced, and the whole is stirred up at intervals for two or three hours till no more solid matter can be felt. The outer beaker is next warmed to 90° C. for ten or fifteen minutes in order to bring certain sparingly soluble iron compounds into solution; the mass is allowed to settle, and filtered. The residue is first washed with water as long as the filtrate gives an iron reaction, next with a boiling solution of ammonium tartrate containing a large quantity of free ammonia to dissolve basic salts precipitated during filtration, and finally with water again; but if the original metal is rich in carbon, the residue should be rinsed off the paper, and extracted several times with the tartrate to ensure solution of iron carbide and phosphate. The slag is then ignited, and freed from silica with sodium carbonate solution. Allowance must be made for the conversion of the ferroso-ferric oxide into ferric oxide during ignition. It is not advisable to dissolve the bromine in any soluble bromide, as the energy of attack is thereby seriously diminished. F. H. L.

Estimation of Molybdenum in Iron. E. Döhler. (*Chem. Zeit.*, 1900, xxiv., 537.)—Between 5 and 20 grammes of borings are dissolved in 100 c.c. of nitric acid, and the solution is evaporated first on the water-bath and then on sand, without igniting the residue, till the acid is entirely driven off. The mass is taken up in 100 c.c. of 1·19 hydrochloric acid, and heated on the water-bath, with repeated additions of water, until not more than 10 c.c. of free acid remain behind. It is next diluted and filtered into a large flask, where the solution (about 1,000 c.c. in volume) is precipitated with sulphuretted hydrogen with the usual precautions used in precipitating molybdenum. After standing covered for twelve hours, the precipitate is filtered and washed five or six times with cold sulphuretted hydrogen-water containing a little hydrochloric acid; the filtrate is concentrated to half its bulk, and submitted to the action of sulphuretted hydrogen a second time. Usually the whole of the molybdenum is recovered at the first precipitation; but should the second current of gas produce darkening of the liquid, it should be set aside for ten hours, after thorough saturation, and handled as before. The filter or filters bearing the molybdenum sulphide are then perforated, and the precipitate washed into the

original flask with hot ammonium sulphide and hot water. If any residue on the paper is soluble in dilute hydrochloric acid, it consists of iron, and may be neglected; but if it is coloured brown it contains molybdenum. In this case the filter must be incinerated at a low temperature, and fused with 2 parts of sodium carbonate and 1 of sulphur; the melt is taken up in water, filtered into the main sulphide solution, which is heated for two or three hours in order to dissolve the whole of the molybdenum. This liquid is filtered, any residue being well washed and fused as above, and finally the solution is heated almost to the boiling-point, when it is mixed cautiously and gradually with an excess of 1·124 hydrochloric acid. The sulphuretted hydrogen liberated is driven off, the precipitate collected on a paper tared at 120° C., washed till the chlorine reaction disappears, and dried at 120° to constant weight. The crude molybdenum sulphide is then transferred as completely as possible to an agate mortar, thoroughly mixed up, and an aliquot portion is ignited gently in a current of hydrogen till the weight is constant, considering the product as pure MoS_2 with 60·00 per cent. of Mo.

The original silica precipitate, which also contains the graphite of cast iron, includes the whole of the tungstic acid from tungsten-steels; but provided it be efficiently washed, and the proportion of silicon and graphite together do not exceed 1 per cent., it will be wholly free from molybdenum. F. H. L.

Determination of Nickel in Nickel Ores. A. C. Langmuir. (*Journ. Amer. Chem. Soc.*, vol. xxii. [2], pp. 102-106.)—Where a small amount of nickel is to be separated from a large amount of iron, the following method is claimed to be superior to all others in use:

One gramme of the ore is heated with 15 c.c. of concentrated nitric acid and 1 to 2 c.c. of liquid bromine until the latter is expelled, the liquid being then evaporated almost to dryness. This treatment prevents the formation of a sulphur bead. After expelling the excess of nitric acid, about 50 c.c. of concentrated hydrochloric acid are added, and the liquid slowly boiled down to about 5 c.c., the nitric acid being driven off, and any nickel residue insoluble in that acid brought into solution.

The solution is made up to 250 c.c. with hot water, and treated with hydrogen sulphide to remove copper. The filtrate, after expulsion of the precipitant by boiling, is carefully treated with nitric acid to oxidize the ferrous iron, excess being avoided, and is then made alkaline with ammonia after a slight cooling. The precipitate is washed with hot water, and the filtrate boiled down.

The nickel in the ferric hydroxide is recovered by dissolving the latter in warm dilute hydrochloric acid, concentrating till pasty, treating with 10 c.c. of hydrochloric acid (specific gravity 1·10), warming gently, and agitating for five minutes or more with 40 c.c. of ether in a separating-funnel, the ferric chloride being gradually dissolved by the ether, and leaving nickel, aluminium, cobalt and manganese, as chlorides in the aqueous solution. To insure complete removal of the iron, the ether treatment should be repeated.

The aqueous solution and the (hydrochloric acid) washings of the ethereal

solution are boiled to drive off the dissolved ether; enough bromine to colour the liquid strongly is then added, followed by ammonia, and the solution boiled to throw down iron, aluminium and manganese, the precipitate being dissolved in hydrochloric acid, and again treated with bromine-water and ammonia.

The filtrates and washings being added to the concentrated filtrate from the first ferric hydroxide precipitate, the liquid (containing the whole of the nickel) is concentrated to 100 c.c., or till separation of ammonium chloride begins, the latter being then destroyed by boiling nearly to dryness with 50 c.c. of concentrated nitric acid. This acid is next expelled by heating with 10 c.c of concentrated sulphuric acid, the cooled liquid diluted with 100 c.c. of cold water, boiled, and neutralized with ammonia, 25 c.c. of which is then added in excess; and, after dilution to 200 to 250 c.c., the liquid is electrolyzed with a current of about 1·2 ampères, the deposition of the nickel being generally complete in three hours. It is considered probable that this ether method would be serviceable in the separation of zinc, or uranium, from iron. C. S.

Colorimetric Estimation of Nickel in Steel. R. Fieber. (*Chem. Zeit.*, 1900, xxiv., 393.)—When nickel steel is dissolved in nitric acid, the iron precipitated with ammonia, and the liquid filtered, the solution exhibits a blue colour proportional in intensity to the amount of nickel present, while the tint is not modified by any other constituent of the metal, large quantities of chromium excepted. To prepare a set of standards, or to estimate the nickel in any particular sample, 6 grammes of steel are dissolved in 1·2 grammes nitric acid; the solution is concentrated as much as possible, brought into a 200 c.c. flask, and the iron thrown down with ammonia. When the whole is cold it is diluted to the mark, shaken well, and filtered. Estimation is performed by comparison, diluting the experimental solution, if necessary, till it matches one of the standards. The standards are best kept in 200 c.c. flasks; they remain unchanged in colour indefinitely, even if exposed to light. An accuracy of 0·05 per cent. is quite possible, even with steels containing less than 1 per cent. of nickel. F. H. L.

NOTE.—The process is not available on account of the variation in colour of nickel solutions due to small alterations in the experimental conditions. B. B.

On the Separation of Nickel and Iron with Ammonia. F. Ibbotson and H. Brearley. (*Chem. News*, vol. lxxxi., pp. 193, 194.)—The defective results apparently obtained by some workers are attributed by the authors to the absorption of the ammoniacal nickel solution by the ferric hydroxide precipitate, and can be improved by adding ammonium salts before the ammonia, but in larger proportion than necessary to form the corresponding ammonium-nickel salts. A better way, however, is to substitute potassium cyanide, the acid solution of the sample being first treated with ammonia to neutralize the free acid. A slight excess of standard potassium solution is then added, the whole is poured into an excess of ammonia, and an aliquot portion of the filtrate is titrated with silver nitrate in the usual manner.

The aforesaid absorption of nickel by ferric oxide can be prevented by slightly

acidifying the solution and heating it to boiling-point, the ferric hydroxide first precipitated being easily dissolved by the undecomposed ferric chloride. The liquid soon becomes turbid; and from this point onwards the ammonia used should be very dilute, the precipitation of the iron being complete with even less ammonia than is needed to produce a permanent turbidity when the neutralization is effected in a cold solution. The main point is to keep the solution acid throughout, since if it be ever so slightly alkaline the separation is imperfect. C. S.

The repeated use of the Double Chloride of Copper and Potassium for the Solution of Steel or Iron in estimating Carbon. G. W. Sargent. (*Journ. Amer. Chem. Soc.*, vol. xxii. [4], pp. 210-213.)—The tedious method of recuperating the copper-potassium chloride solution by aeration or electrolysis may be replaced by direct chlorination. Three quarts can be treated per diem, and, after standing overnight and being filtered, the solution is free from the odour of chlorine, and has almost recovered its original colour, in addition to which it forms a more energetic solvent than when freshly prepared. The solution recuperated in this manner after each time of use can be re-used a number of times, the eleventh solution and filtration being accomplished almost as rapidly as the first. About 1 pound of manganese dioxide and $1\frac{1}{2}$ pounds of hydrochloric acid will recuperate 3 quarts of the double chloride. C. S.

Potentials of Copper Electrodes in Solutions of Copper Precipitates. C. Immerwahr. (*Zeit. anorg. Chem.*, 1900, xxiv., 269-278.)—The experiments were carried out with the object of obtaining a measure of the solubility of the different precipitates in solutions of the precipitant. For details of the method, see Küster (*Zeit. anorg. Chem.*, xxiv.). The following table gives the results with the principal precipitates. The electrolyte in each case contained a gramme-molecule of potassium in addition to the precipitant. The concentrations are in gramme-molecules per litre. In the last column are the potentials between the electrodes and the electrolytes :

Cu Salt.	Electrolyte.		Concentration of Cü-ions.	Potential.
	Formula.	Concentration		
Acetate in saturated solution	$Na{\cdot}C_2H_3O_2$	0·05	$6{\cdot}6 \times 10^{-2}$	−0·580
$CuCO_3$	K_2CO_3	0·046	$4{\cdot}2 \times 10^{-6}$	−0·456
$Cu(OH)_2$	$Ba(OH)_2$	0·00016	3×10^{-8}	−0·393
$Cu(OH)_2$	$Ba(OH)_2$	0·025	5×10^{-9}	−0·370
CuO precipitated from hot solution	$Ba(OH)_2$	0·025	$1{\cdot}8 \times 10^{-11}$	−0·298
CuO prepared by igniting $Cu(NO_3)_2$	$Ba(OH)_2$	0·025	$1{\cdot}2 \times 10^{-19}$	−0·056

Attempts were also made to obtain values with CuS in solutions of H_2S, NH_4HS and NaHS. The copper electrodes were, however, attacked by the electrolytes.

Similar experiments were carried out with solutions of copper, in which the formation of Cü-ions was prevented by a reagent. Thus, in a solution of Rochelle salt containing copper tartrate the concentration of Cü-ions was $2{\cdot}3 \times 10^{-9}$. In

solutions of copper in ammonia the results varied from $1{\cdot}5 \times 10^{-8}$ to 9×10^{-17}. In solutions of copper cyanide in potassium cyanide, they varied from 7×10^{-21} to $3{\cdot}5 \times 10^{-44}$. In the last two cases the potential altered on standing in consequence of the formation of cuprous salts.

The potential of solutions of some cuprous salts was also measured. The data do not exist to calculate the true concentration of the ions. In the following table they are compared to CuCl = 1 :

Cu Salt.	Electrolyte.		Concentration of Cu-ions.	Potential.
	Formula.	Concentration.		
CuCl	HCl	0·05	1	−0·465
CuBr	KBr	0·05	$9{\cdot}3 \times 10^{-2}$	−0·404
CuI	KI	0·05	2×10^{-5}	−0·187
CuCNS	KCNS	0·05	$2{\cdot}4 \times 10^{-7}$	−0·074

At present it is not possible to calculate the actual amount of precipitate dissolved. A. M.

Estimation of Alkali-Metal Carbonates in the Presence of Bicarbonates. **F. K. Cameron.** (*Amer. Chem. Journ.*, 1900, 23, 471-486.)—The solution is titrated with a standard solution of potassium hydrogen sulphate, using phenolphthalein as indicator :

$$Na_2CO_3 + HKSO_4 = HNaCO_3 + NaKSO_4.$$

The products of the reaction are neutral to the indicator, whereas the potassium hydrogen sulphate is strongly acid to it. It is essential that the sulphate solution be run into the carbonate at a rate not exceeding 2 drops a second until the colour just disappears. The liquid meanwhile must be shaken continuously and vigorously.

After the carbonate has been estimated chloride may be determined in the same solution. To do this a few more drops of the sulphate solution are added, and then the chloride is determined by means of silver nitrate solution in the usual way, using potassium chromate as indicator. A. M.

Gas-Volumetric Estimation of Nitrites in Presence of Nitrates or other Soluble Salts. **J. Gailhat.** (*Journ. Pharm. Chim.*, 1900, [6], xii., 9; through *Chem. Zeit. Rep.*, 1900, 207.)—When a neutral solution of a metallic nitrite is mixed with an excess of a neutral boiling solution of an ammonium salt, the following reaction occurs : $NH_4Cl + M'NO_2 = N_2 + 2H_2O + M'Cl$, half of the evolved nitrogen coming from the nitrite and half from the ammonia. The apparatus which Schloesing has recommended for the gasometric estimation of nitrates is fitted with a delivery-tube made entirely of glass, and provided with a stopcock. The flask is two-thirds filled with a saturated solution of ammonium chloride, and the air is expelled by boiling; 10 c.c. of the nitrite solution (5 or 10 grammes per litre) are placed in the funnel, admitted slowly to the flask, the nitrogen is collected and measured in a graduated tube. F. H. L.

Use of Iceland Spar for Standardizing Hydrochloric Acid. H. Thiele and R. Richter. (*Zeits. angew. Chem.*, 1900, 486.)—It has been found that when normal hydrochloric acid is in process of standardization, the molecular weights of sodium carbonate and calcium carbonate being taken at 106·1 and 100 respectively, the strength of the acid, if Iceland spar is adopted as the standard, is represented to be 0·25 per cent. higher than by the silver chloride method, and 0·2 per cent. higher than by the use of sodium carbonate, sodium carbonate and silver thus differing between themselves only by 0·05 to 0·1 per cent. The purity of the spar was then examined, and it was found to contain by gravimetric analysis scarcely 0·02 per cent. of foreign matter (made up principally of the metals with insoluble sulphides). Insignificant traces of strontium, but no barium, were discovered spectroscopically, and it was also ascertained that even after mere drying in the desiccator over potassium hydroxide and sulphuric acid, Iceland spar could not contain any bicarbonate. Rosolic acid had been employed as indicator, but it was found that subsequent addition of methyl-orange showed the same end-point in titration.

The anomaly therefore remained unexplained; but after the above article was written, F. W. Richards published his last researches on the atomic weight of calcium, which make the molecular weight of the carbonate 100·13. Allowing for the 0·02 per cent. of impurities in the Iceland spar, its effective molecular weight in titration thus becomes 100·19; and so all the figures obtained on standardizing hydrochloric acid by the various processes agree excellently. F. H. L.

The Heat- and Acid-Resisting Power of Asbestos. E. van der Bellen. (*Chem. Zeit.*, 1900, xxiv., 392.)—The author has analysed two typical samples of asbestos, and has determined their melting-points by comparison with Seger cones and by the use of a Le Chatelier pyrometer, also testing their power to withstand boiling with a large excess of 1·124 hydrochloric acid:

	Anhydrous Asbestos.	Hydrated Asbestos.
SiO_2	56·79 per cent.	42·09 per cent.
SO_3	0·59 ,,	—
MgO	20·48 ,,	42·16 ,,
FeO	—	1·72 ,,
Fe_2O_3	8·03 ,,	—
Al_2O_3	3·85 ,,	—
CaO	1·00 ,,	0·29 ,,
K_2O (total alkalis as) ...	5·93 ,,	0·32 ,,
H_2O	3·73 ,,	13·59 ,,
Molecular constitution ...	4-5SiO_2, (3MgO + Fe_2O_3 + Al_2O_3 + K_2O), H_2O.	2SiO_2, 3MgO, 2HO.
Ratio of acid to bases ...	1·403 per cent.	0·660 per cent.
Ratio of bases to acid ...	0·608 ,,	1·550 ,,
Melting-point	1150° C.	1550-1570° C.
Solubility in acid ...	5 per cent.	57 per cent.

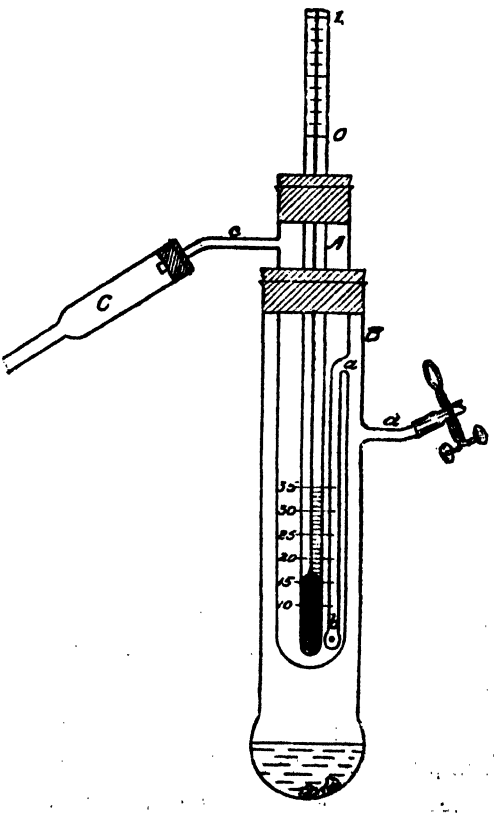
F. H. L.

Asbestos and Chrysotile. C. Reidemeister. (*Chem. Zeit.*, 1900, xxiv., 537.)—Referring to Van der Bellen's article on "Asbestos" (see preceding abstract), the author points out that the word "asbestos" is unfortunately in regular use in trade to designate two substances which are physically similar, but mineralogically and chemically are quite different in character. One of these, containing the higher proportion of silica, is true asbestos; but although it is found in many localities, it is very difficult to procure commercially. The other material, with the lower proportion of silica and the higher melting-point, which is commonly called asbestos as well, is nothing but chrysotile, a fibrous variety of serpentine. This is the substance exported from Canada, and it ought to be described as "Canadian asbestos," or, as Geinitz has suggested, "chrysotile-asbestos."

[*Cf.* Watts' (original) "Dictionary of Chemistry," v., 236.] F. H. L.

APPARATUS.

An Apparatus for Determining Molecular Weights by the Boiling-point Method. H. N. McCoy. (*Amer. Chem. Journ.*, vol. xxiii. [4], pp. 353-360.)—The apparatus shown in the drawing consists of a glass vessel *A*, 2·7 centimetres wide and 20 centimetres long, graduated up to 35 c.c., and fitted with a thermometer. Within, *A* is provided with a narrow glass tube *ab*, opening to the exterior at *a*, 7·5 centimetres from the mouth of *A*, and closed at the bottom except for five small holes *b*. *A* is also connected with a Liebig condenser *C* by a branch tube 2·5 centimetres below the mouth, and is surrounded by the glass jacket *B*, 22 centimetres long by 4 centimetres wide, slightly enlarged at the bottom. About 7 centimetres from the mouth of *B* projects a vent branch tube *d* closed by a rubber tube and pinchcock.

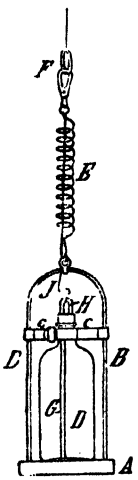


The boiling-point of the pure solvent is determined by placing 50 c.c. in the jacket *B* and 12-16 c.c. in *A*, a small piece of tile being inserted in *B* to prevent bumping. Heat being applied, the vapour produced in *B* passes through *ab* and boils the solvent in *A*, the heat being adjusted so that slow distillation occurs. When the thermometer is constant to within ·001° in thirty seconds the true boiling-point is reached. After opening the tube *d* the flame may be withdrawn and the

substance under examination introduced into *A*, whereupon heat is again applied and the boiling-point of the solution determined as before, the volume of liquid [illegible] being read off after removing the thermometer. The accession of solvent in the inner tube by condensation of vapour from *B* enables readings to be taken at increased degrees of dilution.

The molecular weight is calculated by the formula $M = \frac{W \cdot T}{\Delta \cdot V}$, wherein W = weight of substance, Δ = elevation of boiling-point, and V = volume of solution. The factor T, which is a constant differing for each solvent, is obtained by dividing the corresponding values of the old formula by the specific gravity of the solvents at their boiling-points, the following being the values for a few ordinary solvents: Alcohol, 1560; ether, 3030; chloroform, 2600; benzene, 3280; carbon disulphide, 1940; acetone, 2220; aniline, 3820; water, 540.

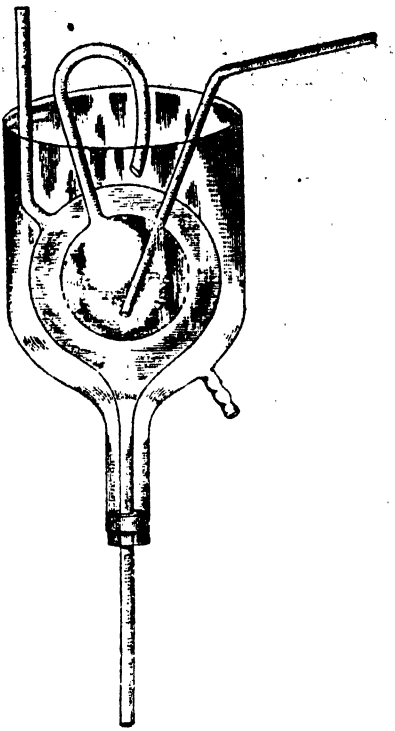
The method is quick and easy, two determinations with different dilutions being obtainable in less than half an hour, and good results are furnished without tedious precautions being required. C. S.



Apparatus for taking Samples of Water beneath the Surface. **F. C. G. Müller.** (*Zeits. Angew. Chem.*, 1900, 388.)—In this apparatus the bottle D is fitted with a rubber cork having two holes, which are simultaneously closed by the U-shaped glass rod H. H is connected directly to the base of the swivel F, whereas the apparatus as a whole is carried by the spring E. A being a lead plate of suitable weight, a sudden jerk on the line lifts H out of the cork, and permits water to fill the flask. F. H. L.

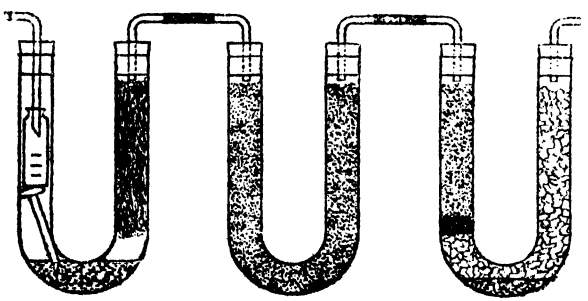
Enamelled Reagent Bottles. **J. Sebelien.** (*Chem. Zeit.*, 1900, xxiv., 538.)—Some years ago enamelled reagent bottles were to be obtained on which the white label was formed of a leadless glaze that would bear immersion in ammonium sulphide for twenty-four hours without discoloration, the black letters consisting of an iridium black, which was capable of resisting strong hydrochloric acid for the same period of time. These bottles appear to be no longer manufactured, and the present articles are made of a more fusible glass, which necessitates the use of a more fusible enamel containing lead, and this is not only blackened by ammonium

...ible, but, being soluble in HCl, allows the lettering, even if it be iridium, to scale off. The author, therefore, considers bottles with etched lettering on a ground of white enamel to be distinctly preferable. When the white lead enamel becomes black owing to the action of sulphuretted hydrogen, the colour may be completely restored by plunging the whole bottle into ordinary hydrogen peroxide solution for about half an hour. F. H. L.



New Upright Condenser. Peters and Rost. (*Zeit. angew. Chem.*, 1900, 28, 688, 689.)—The condensing water passes into the bottom of the inner bulb, passes out through the top, and plays over the surface of the outer bulb. The vapour, which passes up between the two bulbs, is thus exposed to a large condensing surface. The condensation is aided by the evaporation of the water as it passes over the outer bulb. A. M.

Absorption Apparatus for Elementary Organic Analysis. F. G. Benedict. *Amer. Chem. Journ.*, vol. xxiii. [4], pp. 323-334.)—For the absorption of moisture the author recommends the apparatus shown in Fig. 1.



A small glass bottle, to replace the condensing bulb, is inserted in the limb of a $\frac{5}{8} \times 5$ in. U-tube, where it is supported on a short length of glass rod with flattened head, leaving round the bottle an annular space of $1\frac{1}{2}$-2 millimetres, for the passage of the gases escaping from the bottle after the excess of moisture has condensed therein. The other limb of the tube contains a plug of coarse glass-wool, extending to near the bend; through this wool is poured enough sulphuric acid to just seal the bend, leaving a space of about 1 centimetre between the wool and acid to prevent the latter creeping into the carbon-dioxide absorbers, and permit the isolation of the gas bubbles. Most of the water vapour is retained by the seal, and the final traces by the glass wool.

C. S.

REVIEWS.

Traité des Altérations et Falsifications des Substances Alimentaires ("Treatise on the Deterioration and Adulteration of Food"). A. Villiers and E. Collin. Published by Octave Doin, Paris. 1900. 1173 pp., with 633 illustrations; no index. Price 20 francs.

The chief impression left after a perusal of this work is that the conditions under which the French chemist works differ materially from those which affect the English public analyst. His functions to a large extent combine those of public analyst and medical officer of health, and the law, or rather laws, under which he works appear to admit of more definite statements than our own Food and Drugs Acts.

The work is divided into eleven sections, treating of Farinaceous Foods, Stimulants (Cocoa, Coffee, etc.), Condiments, Vegetables, Animal Foods, Dairy Products, Fats and Oils, Sugars, Fermented Liquors, Water, and Preservatives and Colouring Matters. The subjects are treated in a very thorough manner, and the microscopical portions of the book, which occupy more space than is usual in works on food analysis, are excellent; the plates are clear, well drawn, and bring out the salient points in a most distinct manner, and cannot fail to be of great value. The chemical portions, on the other hand, are weak; not that they are incorrect—few points being noted to which real exception can be taken—but they are in but few instances up to date.

The authors appear to have drawn their information almost exclusively from French sources, and the many processes devised in England, Germany, and elsewhere do not seem to have yet filtered through to them, or, if they have reached them, are incorrectly described; thus, the method called by the name of Reichert-Meissl-Wollny contains most of the defects that Wollny pointed out, and guarded against.

A good deal of space is devoted to biological investigations. Good descriptions of the methods of detecting tuberculosis, actinomycosis, and trichinosis in meats occur; the detection of faults of wine is well treated of; and the bacterioscopic examination of water is described. While the discrimination between *B. typhosus* and *B. coli communis*, and the significance of these organisms in water, is ably discussed, no mention is made of *B. sporogenes enteritidis*, nor even of the method of concentrating the micro-organisms by filtration through a reversed Berkefeld filter. The chemical examination of water is based on the methods employed at Montsouris, which were devised for scientific investigation, and, as they require something like 20 litres if carried out as described, can hardly be considered practical.

Some of the methods will be new to English public analysts. The fact that the extensive adulteration of jams with agar is allowed to pass unchallenged in England would show that the method described for the detection of this substance is not generally known.

The analyst who adds this work to his library will be well repaid by the excellence of the plates, even if he does not profit much by the descriptions of chemical processes; the chemist who works by it will be enabled to arrive at correct conclu-

work as to the composition of foods, though frequently more laboriously than if he used methods commonly employed in England; the reader will certainly have his mind broadened by viewing the subject from a French point of view.

The authors, in the preface, recognise the necessity for further research, and propose to fill a portion of the void; if they could be persuaded to study the pages of the ANALYST and other foreign journals, and combine the information they glean with their own researches, the value of future editions would be enhanced.

H. D. R.

COMMERCIAL ORGANIC ANALYSIS. By ALFRED H. ALLEN. Third Edition. With revisions and additions by the author and Dr. HENRY LEFFMANN. Vol. II., Part II. Hydrocarbons, Petroleum and Coal-tar Products, Asphalt, Phenols and Creosotes. London: J. and A. Churchill, 1900. Price 14s.

This volume is the second instalment of the third edition of vol. ii. of Mr. Allen's well known and, to analysts, indispensable work. Owing to the rapid growth of subject-matter, it has been found necessary to split the volume up into parts. Part I., already published, is devoted to the fixed oils, fats, waxes, and products derived from them. Part II., the present volume, treats of hydrocarbons, tars, bitumens, petroleum, shale and coal-tar products; and Part III., in preparation, will comprise the terpenes, essential oils, camphors, and resins. Now that this work consists of no less than eight separate volumes, it may be suggested to the publishers that it would be a great convenience if the main contents of each were lettered on the back.

In the revision, especially of the first half of the volume, Mr. Allen has again had the assistance of Dr. Leffmann; the latter half, particularly the section on "phenols," has received much of his own personal attention. The revision has necessarily been extensive, and the text has been largely rewritten. Space has been economized by the omission of some portions of the text of former editions which more properly belong to works on theoretical organic chemistry. The hydrocarbons are systematically arranged and named according to the most modern system. Acetylene, which, when the last edition was published, was of merely theoretical interest, now forms the subject of a separate sub-section, which, however, is rather unduly condensed. A very complete account is given of asphalt, the analysis of which has of late years attracted much attention in the United States. The best-known methods are described, but it is unsatisfactory to note that all are more or less arbitrary, and do not even give comparable results on the same sample. Asphalt paving is extensively used in American cities, and some official specifications are given, showing the composition of the mixtures used.

The subject of lubricating oils, which in the second edition was treated partly under Fats and partly under Hydrocarbons, is now collected into a single section, which is a better arrangement. Under Viscometry, or Viscosimetry, a description is given of Doolittle's torsion viscometer, which is said to be much used in American laboratories. The chief advantage claimed for this instrument over the common form of jet viscometer is that the results obtained are not influenced by differences in the specific gravities of the liquids tested. Pure cane-sugar solutions of arbitrary strength are used for standardizing. Mixtures of glycerol and water of known viscosity would, we

think, be preferable; and if it could be shown that the indications of this instrument are in proportion to the true viscosities of all liquids tested by it, both those of low as well as of high viscosity, it would deserve to come into more general use, and to replace the imperfect jet viscometers at present in vogue. It is rather strange that the Abel apparatus should be described as the best for determining the flashing-points of lubricating oils (p. 136), seeing that the Pensky-Martens and Gray testers (both modifications of the Abel) have been specially designed for the purpose. The Abel apparatus is usually put together with soft solder, which melts at 170° to 180° C. With regard to the statement on p. 146, that paraffin dissolves in warm fixed oils, and does not separate on cooling, it may be pointed out that some kinds of mineral lubricating oil mix with rape oil perfectly when warm, but on cooling or standing solid hydrocarbons are apt to separate, and cause troublesome deposits in lubricators.

The latter half of the volume, on cyclic hydrocarbons from coal-tar and their derivatives, has been very thoroughly revised and largely added to. Special tests are given for many benzene derivatives now used as drugs, food-preservatives, or disinfectants. Mr. Allen has largely added to the data concerning phenols, phenolic disinfectants, and wood-tar creosote; but no reference is made to the disinfectant known as "Izal," obtained from coke-oven tar. On p. 254 an excellent method is given for estimating the phenols in disinfecting powders with non-alkaline base, which may be still further simplified by dispensing with the use of Soxhlet's extractor. Percolation with small quantities of ether about three times in a tapped cylindrical funnel, having a loose plug of asbestos at the bottom, extracts the whole of the phenols in about half an hour. Powders with lime base may be analysed by boiling them with 10 per cent. solution of caustic soda, making up to a known weight, settling, decanting or drawing off an aliquot (weighed) portion of the clear liquid, concentrating, and throwing up the phenols with acid. This saves the tedious process of neutralizing the powder with strong sulphuric acid as described on p. 255.

Dr. Leffmann has added at the end of the volume an appendix describing some new forms of extracting and drying apparatus, also tables of comparison of Beaumé degrees and specific gravities of liquids lighter than water, and a useful table of comparisons of thermometric degrees. The text contains very few errata, one of the most puzzling being the reference on p. 252, first line, to "p. 559," which is a page of the second edition, and should be "p. 301." Also, on p. 255, in the first line of the third paragraph, the reference to the obsolete "distillation process" is a similar slip. In the figure on p. 79, the arrow referred to on p. 80, line 2, is missing. "Gasolene" (or gasoline ?), on p. 22, line 9, is a misprint for probably "rhigoline."

Chemists of the present generation are deeply indebted to Mr. Allen, not only for having equipped them with such a valuable work as "Commercial Organic Analysis," but for devoting the best years of his life to the labour of keeping the work up to date. He is to be congratulated upon having secured the assistance of able collaborateurs in carrying on the work so ably begun.

L. A.

ERRATA.

This volume, p. 148, line seven from bottom, *for* "325° C.," *read* "315° C."; line twelve from bottom, *for* "350°" *read* "350° C."

THE ANALYST.

OCTOBER, 1900.

PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS.

THE DETERMINATION OF OXYGEN IN COPPER BY IGNITION IN HYDROGEN.

By L. Archbutt, F.I.C.

(*Read at the Meeting, June* 6, 1900.)

In consequence of some anomalous results obtained during the autumn of 1893 in determining oxygen in commercial copper by Abel's silver nitrate method, which I had up to that time employed with confidence, my attention became directed to a paper on "The Analysis of Copper," by W. Hampe (*Zeits. für anal. Chem.*, 1874, pp. 176-234), in which the estimation of oxygen is directed to be made by heating the copper to redness in a current of hydrogen. The unfavourable results previously obtained by Abel (*Journ. Chem. Soc.*, 1864, pp. 166-172), which led him to conclude "that correct indications of the proportion of oxygen existing in a specimen of copper cannot be obtained by treatment of the metal with hydrogen," did not encourage further experiment in the same direction; but the experimental results reported by Hampe in his paper were so satisfactory that I determined to make a careful trial of his method, of which the following is a description:

Estimation of Oxygen (*Hampe*).

The clean copper is converted into filings with a not too coarse file, the filings are passed through a hair sieve in order to separate any coarse particles, fragments of iron are removed by a magnet, and the powdered copper is washed either with a dilute solution of caustic potash, followed by water, or with pure alcohol or ether, in order to remove grease. The purified copper is dried. For the reduction a bulb-tube of Bohemian glass is used, drawn out at both ends. This is connected with a drying apparatus, through which a current of air is passed from a gasometer. The empty bulb-tube is first heated in the stream of air, allowed to cool, and at once closed with indiarubber stoppers. The tube having been weighed, 30 to 50 grammes of the prepared copper are placed in the bulb, the tube is again weighed, and a current of dry, air-free CO_2 is passed through it. This CO_2 is prepared from marble and hydrochloric acid, and is purified by passing through vessels containing sodium carbonate

and silver nitrate, and dried by passing over sulphuric acid and calcium chloride. The apparatus is set in action two hours previous to use, in order to drive all air out of the purifying train.

After the stream of CO_2 has passed over the copper for about five minutes, a very moderate heat is applied, in order to expel every trace of superficial moisture. With many coppers which contain arsenic, too strong heating will result in the formation in the bulb, or just beyond it, of a sublimate of As_4O_6. After cooling in CO_2, a stream of dry air is immediately passed through the bulb-tube to displace this gas, and the tube is again stoppered and weighed. The weight is, usually, only a few milligrammes less than the previous weight.

A very slow stream of hydrogen is now conducted over the copper, which is heated at first gently, and afterwards to a red heat, which temperature is maintained for about fifteen minutes. During the heating water is formed, and by impure copper there is also formed a black sublimate containing arsenic, antimony, and lead, so that the end of the tube must be long enough, and the stream of hydrogen slow enough, to insure the whole of this sublimate being retained within the tube.

During the formation of water there is noticed, with commercial copper, an evolution of H_2S. The formation of this gas, already observed by Dick, but not explained, can be accounted for in the author's (Hampe's) opinion only by the presence in the copper of sulphur dioxide, a part of the sulphur of which is converted into H_2S. Cuprous sulphide cannot exist in copper along with oxygen compounds, and would not be decomposed by heating in hydrogen. To retain the escaping H_2S, the bulb-tube must be connected with several small flasks containing silver solution or alkaline lead solution, and the sulphur can then be estimated by known methods. Arsenic and antimony cannot be found in the precipitate. After the copper has completely cooled in the stream of hydrogen, and this gas has been displaced by dry air, the tube and copper are again weighed. As control, a duplicate experiment is desirable. The mean loss of weight, less the weight of volatile sulphur, is oxygen. The correction for sulphur seldom exceeds 0·002 per cent. in refined copper. The following results were obtained by Hampe:

A. Synthetically prepared Samples.

	Oxygen Present (per cent.).	Oxygen Found (per cent.).
1. Copper containing copper oxide	0·247	0·237
2. Copper containing "copper mica"	0·262	0·267
3. Copper containing bismuth antimonate, of which the oxygen content must have been from	0·141 to 0·170	0·159

B. Copper from Oker.

	Oxygen per Cent., after correcting for Volatile Sulphur.			
	First Experiment.	Second Experiment.	Third Experiment.	Mean.
1. Tough copper ...	0·117	0·117	—	0·117
2. ,, ,, ...	0·154	0·153	0·165	0·157
3. Dry copper ...	0·806	—	—	0·806

C. Copper from Mansfield.

	Oxygen, per Cent., after correcting for Volatile Sulphur.			
	First Experiment.	Second Experiment.	Third Experiment.	Mean.
1. "Black copper"	0·443	—	—	0·443
2. Dry refined copper	0·727	0·765	—	0·746
3. Tough ("dichtgepoltes") copper ...	0·147	0·162	—	0·155
4. Tough ("zähe gepoltes") copper ...	0·077	0·072	0·077	0·075
5. Overpoled copper	0·045	0·044	—	0·045

In connection with the above analyses of dry copper, it is interesting to note that Dick, in 1856, found about 1·4 per cent. of oxygen in a specimen of copper "in the driest state," which Percy had procured specially for experiment. This result of Dick's was obtained by rolling the copper as thin as possible, cutting the rolled metal into small pieces, exposing these to a current of dry hydrogen at a red heat and weighing in a calcium chloride tube the water formed. Hampe refers to Dick's experiments, but apparently makes no mention of Abel's work in the same direction, which is strange, as Abel tried for a long time to estimate the oxygen in copper by a method similar to the above-mentioned process used by Dick, and came to the conclusion "that although the larger proportion of the oxygen contained in a specimen of copper may perhaps be abstracted during the first two or three hours' treatment, its complete removal from the metal in the form of water requires very long continued exposure to the action of hydrogen." In several experiments a continuous increase in the weight of the calcium chloride tube was observed, which "appeared likely to continue for an indefinite period," and which was attributed partly to traces of atmospheric oxygen obstinately retained by the copper. After numerous failures to obtain satisfactory results, Abel finally abandoned the method as impracticable.

Blount much more recently (*Analyst*, 1896, pp. 57-61) came to the conclusion that the action of hydrogen on copper at any temperature below the softening-point of a glass combustion tube is only superficial, and he has described a method in which the copper is actually fused in a current of hydrogen. Blount found, as Dick had done, that the reduction is accompanied by such violent spirting of the molten copper that an estimation by loss of weight is impracticable; but by conducting the operation in a porcelain tube, and absorbing the water formed in a sulphuric acid drying tube, he has worked out an elegant and successful process. My own experiments, however, and several years' subsequent experience, have convinced me that the reduction takes place easily and completely at a red heat, and that Hampe's process is accurate as well as, when certain unnecessary details are omitted, extremely simple. By the courtesy of Mr. Blount, an opportunity has recently been afforded of proving that the results obtained by the two methods are practically identical. An account of my experiments and of the results obtained may, therefore, be of interest to the Society.

I commenced by following Hampe's directions closely, but arrangements were made for absorbing and weighing the water formed, as a check on the loss of weight undergone by the copper. An apparatus for the supply of dry oxygen-free hydrogen was therefore fitted up, which consisted of: (*a*) Kipp generator, containing Leclanché

zinc rod and dilute pure sulphuric acid (1 : 4); (*b*) a small washing bottle, containing 50 per cent. solution of caustic soda; (*c*) a tower, 210 millimetres high by 46 millimetres diameter, containing pieces of stick soda; (*d*) a combustion tube, containing about 400 millimetres of copper gauze rolled into a close spiral and kept red hot; and (*e*) a U-tube, 300 millimetres long, filled with freshly-fused caustic potash broken into small lumps (as recommended by Dittmar). The hydrogen generated and purified in this apparatus was passed through a weighed drying tube, containing sulphuric acid and pumice, for two hours at the rate of 200 c.c. per hour, and for two hours more at four times that rate. The total volume of hydrogen passed was about 2 litres, and, after displacing the hydrogen remaining in the tube by dry air, the weight of the tube was found to be exactly the same as at the commencement.

The copper was used in the form of bright turnings, small enough to pass through a tube $\frac{1}{8}$ inch in diameter. These were thoroughly washed by decantation with ether to remove grease, and dried by heating on the water-oven and cooling in a desiccator. Finer division of the copper than this is not necessary. Filings may be used if more convenient, but they will contain a more or less considerable admixture of steel from the teeth of the file, which must be carefully picked out by a magnet, and they must be very well washed with ether to remove grease and dirt. There does not appear to be the least necessity to pass the filings through a hair sieve, as Hampe directs.

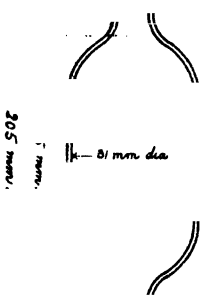


Fig. 1.

Very convenient bulb-tubes for containing the copper are made for me of Jena glass, of the shape and dimensions shown in Fig. 1. They are suspended for weighing by means of a platinum wire hanger with sufficiently large loops at either end to slip easily off and on the tube. These tubes can be used without serious alteration of shape for a large number of operations, in fact, until the glass becomes opaque. During the combustion a black deposit of lead, arsenic, etc., usually forms near the exit end, but is easily cleaned off afterwards with a solution of bromine in hydrochloric acid. The tube is then thoroughly washed with water and dried in the water-oven. The tubes scarcely alter in weight with repeated use. The following are the weights in grammes of three different tubes after being used for three successive combustions:

(1)	(2)	(3)
53·593	48·155	48·654
53·594	48·155	48·654
53·593	48·155	48·654

The clean dry tube, when cold, is wiped outside and allowed to remain supported across a beaker, not in a desiccator, for ten minutes. It is then weighed. One end having been closed by a tiny cork, a small funnel is attached to the other end, and about 30 to 35 grammes of the prepared copper are dropped in. The cork and funnel are detached, the copper is shaken into the bulb, and the tube is wiped and weighed

as before. The weight of copper taken for assay is thus ascertained. I prefer to weigh the tube open at the ends, as I have found that indiarubber stoppers are not only unnecessary, but they are apt to cause errors in the weighings.

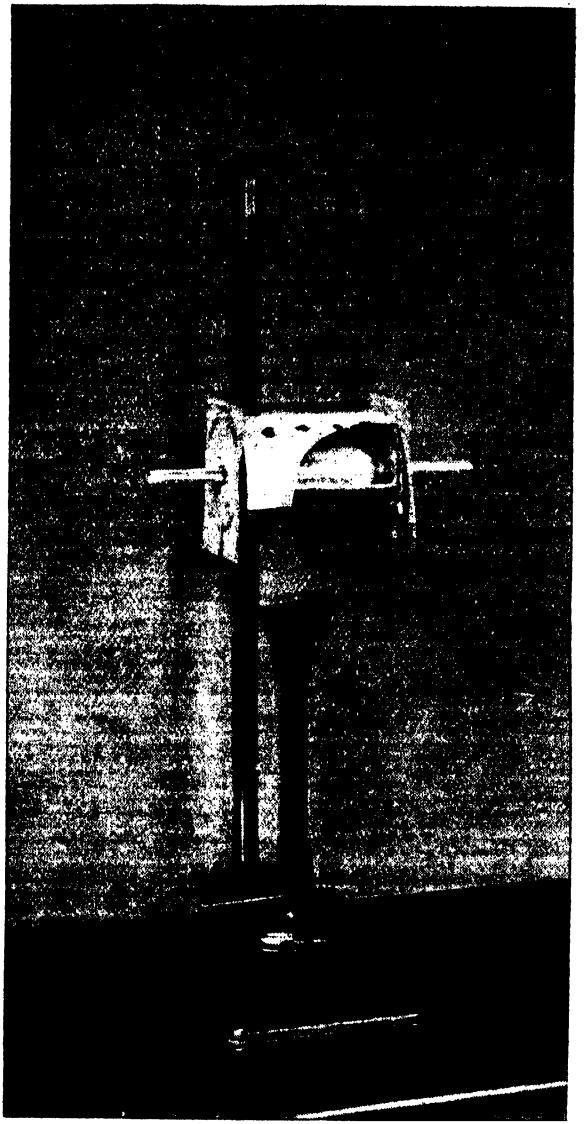


FIG. 2.

The bulb-tube containing the copper is supported on a sheet-iron tray lined with asbestos millboard, a piece of perforated sheet asbestos is bent over the top and kept in shape by an iron-wire frame, and flat pieces of asbestos, perforated in the centre to receive the ends of the bulb-tube, are placed at the sides (see Fig. 2). The bulb, thus

surrounded on all sides by asbestos, is heated by means of a Bunsen burner giving a broad flat flame.

The following three experiments were made with firebox-plate copper containing 0·063 per cent. of arsenic:

COPPER FIREBOX PLATE. A.

	First Experiment.	Second Experiment.	Third Experiment.
Weight of copper taken	29·7526	35·0023	33·5010
Weight of copper and tube before heating ...	58·4844	63·6016	62·5867
,, ,, after heating in CO_2 ...	58·4834	63·6025*	62·5833†
,, ,, after heating in hydrogen ...	58·4354	63·5441	62·5268
Loss of weight in hydrogen	·0480	·0584	·0565
Water absorption tube after	49·1466	49·3808	49·4428
,, ,, ,, before	49·0904	49·3138	49·3808
Weight of water formed	·0562	·0670	·0620
Containing oxygen	·0500	·0595	·0551

* Copper became slightly oxidized at one end. † Heated to redness.

PERCENTAGE OF OXYGEN FOUND.

	By Loss of Weight of Copper.	By Weight of Water formed.
Experiment 1	0·161	0·167
,, 2	0·167	0·170
,, 3	0·169	0·165
Mean	0·166	0·167

The copper remaining after Experiment 1 was transferred to another tube (the first having developed a minute crack) and reheated, with the following results:

	Fourth Experiment.
Weight of copper and tube before heating	58·2991
,, ,, ,, after heating in CO_2	58·3001
,, ,, ,, after heating in hydrogen to full redness for half an hour	58·3000
Loss of weight in hydrogen	·0001

This experiment shows how easily a constant weight in hydrogen is obtained. In fact, by watching the progress of an experiment one soon acquires confidence in the method. As soon as the copper reaches a dull red heat, water begins to condense rapidly in the first cool place, and has to be chased along by a small flame; after heating for about half an hour, it ceases to condense. In the above experiments the heating was continued for about two hours longer, the current of hydrogen being at the rate of 200 to 300 c.c. per hour.

The following experiments were made with two other firebox-plate coppers, "B" containing 0·031 per cent., and "C" 0·485 per cent., of arsenic. In Experi-

ment 7 the heating in CO_2 was carried to rather too high a temperature, and a sublimate of As_4O_6 formed in the end of the bulb-tube. In Experiment 8 no sublimate was formed.

COPPER FIREBOX PLATE B.

	Fifth Experiment.	Sixth Experiment.
Weight of copper taken	32·9989	35·1397
Weight of copper and tube before heating	60·6767	62·8178
,, ,, ,, after heating in CO_2 ...	60·6745	62·8140
,, ,, ,, after heating in hydrogen ...	60·6305	62·7657
Loss of weight in hydrogen	·0440	·0483
Water absorption tube after	49·4910	
,, ,, ,, before	49·4427	
Weight of water formed	·0483	
Containing oxygen	·0429	

	Percentage of Oxygen Found.	
	By Loss of Weight of Copper.	By Weight of Water formed.
Experiment 5	0·133	0·130
,, 6	0·137	
Mean	0·135	

COPPER FIREBOX PLATE C.

	Seventh Experiment.	Eighth Experiment.
Weight of copper taken	34·5900	34·4244
Weight of copper and tube before heating	59·7740	67·2730
,, ,, ,, after heating in CO_2 ...	59·7713	67·2725
,, ,, ,, after heating in hydrogen ...	59·7158	67·2171
Loss of weight in hydrogen	·0555	·0554
Water absorption tube after		46·9477
,, ,, ,, before		46·8869
Weight of water formed		·0608
Containing oxygen		·0540

	Percentage of Oxygen Found.	
	By Loss of Weight of Copper.	By Weight of Water formed.
Experiment 7	0·160	
,, 8	0·161	0·157
Mean	0·1605	0·157

From the foregoing experiments it appears that practically the same results are obtained, whether the oxygen be estimated from the loss of weight of the copper or from the weight of water formed. The former method is much the simpler. It seems hardly necessary to go to the trouble of recovering the trace of sulphur which passes off as H_2S. It can only influence the result in the third decimal place, and such accuracy is not required in the present state of our knowledge.

If the water formed in the combustion is not weighed, there is no need to purify the hydrogen from traces of oxygen. This was not done in Experiment 7, whilst in Experiment 8 it was, and it will be observed that the loss of weight of the copper is practically the same in each case. The tube containing the red-hot copper gauze

RESULTS OBTAINED BY ASSAY FOR OXYGEN OF TWENTY-THREE SAMPLES OF COMMERCIAL COPPER.

Experiment No.	Description of Copper.	Weight taken.	Loss of Weight in CO_2.	Loss of Weight in H.	Equivalent to Oxygen.	Other Particulars. Arsenic.	Other Particulars. Phosphorus.	Remarks.
		Grammes.	Grammes.	Grammes.	Per cent.	Percent.	Percent.	
9	Tube ...	39·1339	0·0000	0·0028 = 0·007		trace	0·099	
10		35·8081	0·0005	0·0032 = 0·009		nil	0·033	
11		35·2848	0·0004	0·0018 = 0·005		nil	0·026	
12	Rod ...	32·9537	0·0004	0·0237 = 0·072		0·377	nil	
13	,, ...	30·4792	0·0003	0·0220 = 0·072		0·074	nil	
14	Firebox plate	33·9440	0·0001	0·0269 = 0·079		0·037		
15*a*	.. ..	33·5455	0·0005	0·0383 = 0·114				Duplicate tests of the same sample.
15*b*	,, ,,	32·0803	0·0005	0·0370 = 0·115		0·510	—	
16		31·6886	0·0006	0·0498 = 0·157		0·268		
17		32·3282	0·0006	0·0230 = 0·071		0·218		
18		34·6286	0·0006	0·0579 = 0·167		0·463		
19		32·0084	0·0006	0·0464 = 0·145		0·034		
20		33·5971	0·0005	0·0380 = 0·113		0·215		
21		35·0629	0·0006	0·0471 = 0·134		0·172		
22		36·7553	0·0001	0·0331 = 0·090		0·013		
23		37·7511	0·0009	0·0733 = 0·194		0·200		
24		36·5867	0·0006	0·0576 = 0·157		0·021		
25		38·6362	0·0008	0·0530 = 0·137		0·181		
26*a*	,, ,,	36·6247	0·0011	0·0494 = 0·135				Duplicate tests of the same sample.
26*b*	,, ,,	35·1720	0·0021	0·0469 = 0·133		0·148		
27		35·8049	0·0000	0·0379 = 0·106		0·234		
28*a*	,, ,,	37·6476	0·0008	0·0413 = 0·110				Duplicate tests of the same sample.
28*b*	,, ,,	40·0071	0·0005	0·0435 = 0·109		0·624	—	
29	,, ,,	35·6995	0·0010	0·0738 = 0·207		0·310		
30	,, ,,	36·2921	0·0008	0·0442 = 0·122		0·102		
31	,, ,,	37·4032	0·0006	0·0386 = 0·103		0·160		

spiral was therefore removed from the purifying train, and the twenty-three samples of copper included in the table on the opposite page were assayed for oxygen thus:

1. The prepared copper was heated in purified, dry CO_2 for half an hour, during which time 500 c.c. of the gas were passed through the bulb, the temperature being high enough to expel moisture, but not high enough to sublime As_4O_6 from the copper.
2. The dry copper was heated to redness in hydrogen, which had been purified from acid vapours and water, but not from traces of oxygen. The temperature was maintained at a red heat for one hour, about 1 litre of gas being passed through the bulb.

I have included in the table the percentages of arsenic and phosphorus found in

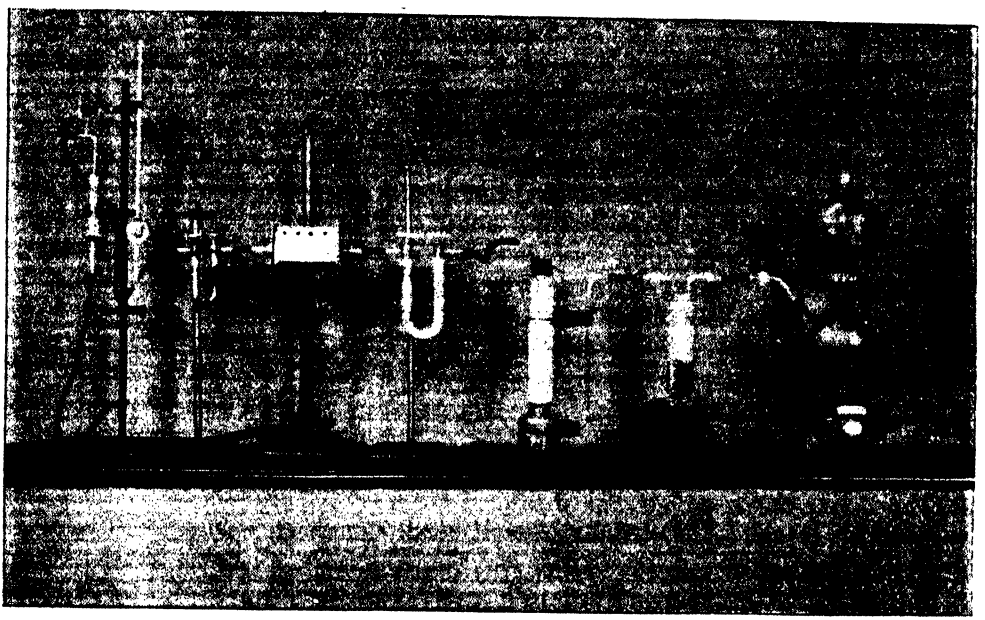


FIG. 3.

these coppers. Copper containing phosphorus is invariably free from oxygen, or contains only traces of that element, and a considerable proportion of the loss on ignition in hydrogen of such copper is probably made up of the traces of oily hydrocarbons which usually distil from copper when heated, however carefully it has been washed with ether.

The loss of weight in carbon dioxide is seen to have been very trifling in all these experiments except one (No. 26*b*). In fact, it was so small that I have now abandoned this part of Hampe's process, and merely wash the copper with ether, and dry it well in the water-oven before heating in hydrogen. Although the water resulting from the combustion in hydrogen is not weighed, a trap of some kind beyond the bulb-tube is necessary, and may conveniently consist of a U-tube containing pieces of glass rod and sufficient sulphuric acid to fill the bend. Fig. 3 is from a photo of the complete

apparatus which I now employ for the combustion in hydrogen. It comprises (1) Kipp generator; (2) washing-bottle, containing caustic soda solution; (3) tower, containing stick soda; (4) U-tube, containing re-fused dry caustic potash in small pieces; (5) bulb-tube, containing the prepared dry copper; (6) U-tube, containing strong sulphuric acid; and (7) pipettes, for measuring the rate of flow of hydrogen.

Process.—The copper (30 to 40 grammes), which must be free from superficial oxide, is thoroughly cleansed with ether, carefully dried, introduced into the dry bulb-tube, and weighed, as already described. The apparatus is joined up, and a current of hydrogen (about 1 litre per hour) is passed through it until the issuing gas burns quietly, which is usually the case after fifteen minutes have elapsed. The current of hydrogen is then reduced by about one-half, and if much arsenic is present in the copper it is still further reduced; the copper is then heated up, with a flame just touching the iron support, until water begins to condense beyond the bulb-tube. If any sublimate of As_4O_6 be observed, the current of hydrogen is made very slow, and the tube is heated more strongly, so as to reduce the oxide to a mirror of arsenic within the tube. Any water condensing between the bulb and the U-tube is chased along by a small flame. Gradually the copper is raised to full redness, and, when water ceases to condense, the current of hydrogen is increased to 1 litre per hour. After continuing to heat the copper thus for one hour, the gas is turned out, but the current of hydrogen is kept up until the copper is quite cold. The bulb-tube is then disconnected, the hydrogen is displaced by aspirating a current of dry air through the tube, and the latter is weighed and the loss calculated as oxygen per cent. of the copper taken.

The following results were obtained by Mr. Blount and myself, working independently on the same specimens of copper. Mr. Blount's determinations were made by fusion of the copper in hydrogen, and weighing the water produced; my results were obtained by simply heating to redness in hydrogen and observing the loss in weight of the copper, as described above. The agreement is sufficiently close to show that both methods give practically identical results. The comparatively large discrepancy in the case of No. 3 sample may be due, Mr. Blount thinks, to the fact that the copper was in rather coarse pieces for his process, and he had to work on comparatively small quantities: the small constant plus error due to the almost inevitable gain of an absorption tube would, therefore, have a larger effect on the final result in this case than it would under the normal conditions of working when the copper is large. The results in the second column were obtained by my assistant, Mr. P. G. Jackson, A.I.C.

Sample Number.	Oxygen per cent. determined by B. Blount.	L. Archbutt.
1	0·18	0·17
2	0·22	0·21
3	0·05	0·003
4	0·03	0·03
5	0·13	0·12
6	0·25	0·25

Discussion.

Mr. Blount said he was glad to find that processes of the same class as the old silver nitrate process might be reckoned as deleted. There was, in fact, only one method of determining the oxygen in copper—namely, by reduction in hydrogen. The only point upon which it was possible to base a discussion was the fact that there was some doubt—or had been until that occasion—as to the practicability, at a temperature short of the fusing-point of copper, of determining by reduction in hydrogen the whole of the oxygen present. In his own experiments he had doubted this so much that he was ultimately led to abandon the notion of reducing at a temperature lower than fusing-point. He felt bound to say, however, that the reduction in hydrogen of the oxides present in copper, when the copper had to be fused, was not an altogether convenient proceeding, and if it were possible, as Mr. Archbutt had shown it to be, to reduce the oxides without raising the copper to its fusing-point, a great step would have been taken in advance. Nevertheless, he (Mr. Blount) could not help thinking that there were certain reasons which would lead one to prefer the method he had ventured to recommend, of fusing the copper in hydrogen, and weighing, not the copper remaining, but the water produced. It seemed evident that the determination of an impurity occurring in small proportion, such as oxygen in copper, must be made more or less by determining the difference between two comparatively large weights—*e.g.*, of the vessel in which the impurity was contained. When the substance constituting the impurity was isolated—as, for instance, in the case of bismuth, the proportion of which in commercial copper was generally under 0·01 per cent.—the errors would be quite tangible; and in the same manner the increase in the weight of the absorption tube in determining the oxygen by fusion of the copper in hydrogen was so small that any error would assume relatively large proportions. But the method of determining the oxygen by difference between the weights of the copper before and after reduction in hydrogen was still more open to error, rendering necessary not only the most minute accuracy in weighing the original mass in the tube and the final mass, but also many precautions against the escape of volatile constituents other than oxygen. There could be no guarantee that arsenic, for example, would remain unvolatilized. The fact that such admirable results were obtained by the author was a testimony to his skill and that of those whom he had trained, rather than to the excellence of the process. In the preparation of the copper for the determination of oxygen by this method it was necessary to subdivide it; and although the author had succeeded in overcoming the difficulties due to contamination from files, drills, etc., it was really unnecessary to face them when one could work satisfactorily upon lumps of copper obtained without any risk of such contamination. From a purely utilitarian point of view, the question of speed was of considerable moment, and it might be mentioned that the method which he had described in 1896 enabled as many as twelve determinations of oxygen in copper to be made in a working day, with the same apparatus and by one operator.

Mr. Jenkins said that he was personally indebted to the author for having introduced this method to his notice several years previously. He thought that Mr. Blount had altogether overemphasized the objections which may be brought

against the method. The machining of the metal into small fragments was easily done, without any contamination with oil, etc., not only in the case of copper, but of steel and other materials. It was shown by the figures given in the paper that the results obtained by absorption of the water were practically the same as those obtained by difference. It would certainly in many cases be more convenient to make the determination by Mr. Archbutt's method than by Mr. Blount's method, seeing that the former did not involve working at so high a temperature, and it was highly satisfactory that Mr. Archbutt and Mr. Blount, working at considerable differences of temperature, should get such closely-agreeing results.

Mr. ALLEN said that he had an extensive experience of the burning of metals in oxygen, which was a process somewhat analogous to the one now under consideration, and he must say that he would give preference to a method in which drillings or finely-divided particles of metal were used. He altogether deprecated the suggestion that there would be contamination by oil from the drill or other tool. There was no difficulty in getting drillings free from oil in the case of steel, and it would be still easier in the case of copper. Mr. Blount, however, had given good reasons for believing the fusion process to be quicker and equally accurate, and therefore the matter was one of convenience rather than of accuracy. He had heard with some surprise that where a sample of copper contained arsenic it was devoid of phosphorus, and *vice versâ*. He would like to hear whether the author could suggest any possible explanation of this very curious fact.

Mr. ARCHBUTT, in reply, said that, after all, it did not matter whether the determination was made by fusion or by loss of weight at a red heat; the material point which he had sought to establish was that both methods gave practically the total percentage of oxygen present. Every sample of copper in which he determined the oxygen, and which he also completely analysed for other elements, was in the form of turnings from a test-bar which was subjected to mechanical tests. It seemed to him that this was a necessity if any information was to be obtained as to the extent to which the chemical composition bore upon the mechanical qualities of the metal. He had found these turnings very convenient to work upon. In obtaining them, as far as possible anything in the way of grease was excluded, but, nevertheless, the precautions were taken of washing the turnings with ether, and of picking out any iron with a magnet. He was glad to gather, from Mr. Blount's remark as to the importance of speed, that engineers and purchasers of copper take so large an interest in the bearing of the chemical composition upon the wearing qualities of the metal, as this would afford the means of obtaining information which really did not exist at present. He did not know that he was in a position to state exactly why phosphorus and arsenic never occurred together in copper, but that was his experience. He had never met with a firebox plate containing phosphorus; but phosphorus was generally present in copper used for making tubes; such copper was practically free from oxygen, and was, he believed, deoxidized by adding phosphorus to it. He had so frequently found phosphorus absent when arsenic was present that he had ceased to test for it in such cases. Arsenical copper always contained oxygen, and such copper could not contain phosphorus.

Mr. BLOUNT entirely endorsed Mr. Archbutt's view, that the sample for analysis

should come from the actual test bar, though it did not follow necessarily that it should consist of turnings.

Dr. Dyer said he believed that small variations in the proportion of oxygen present had considerable effect upon the electrical conductivity of the copper. He would like to ask the author what quantity of oxygen would be expected in good electrical copper, and, more especially, how far variations in the proportion of oxygen affected the strength and durability of the copper for mechanical purposes.

Mr. Archbutt said that he had no experience of the composition of copper for electrical purposes, but there seemed to be a definite relationship necessary between the proportions of oxygen, lead, arsenic, etc., in order to yield a good tough copper, though exactly what that relationship was he could not at present say. He had a number of figures showing a remarkable relationship between the cuprous oxide determined by Abel's method and the arsenic and lead, but exactly what that meant was not yet clear.

A NEW COLOUR REACTION FOR CITRAL AND CERTAIN OTHER AROMATIC COMPOUNDS.

By Herbert E. Burgess.

(*Read at the Meeting, May* 2, 1900.)

In various endeavours I have made to discover a quantitative reaction which could be used for the estimation of citral (especially in oil of lemon), I was led to try the action of mercury salts on aldehydes, with a view to determine the amount of reduction, or, if possible, to weigh the compound so formed. Up to the present time I have been unable to base any satisfactory quantitative method on my results, owing to the fact that many other allied aromatic compounds form salts with the reagent I employ, and also that these mercury compounds are not easily crystallizable, and are probably of indefinite composition and transitory.

I have, however, noticed well-defined colour reactions with many of the aldehydes, alcohols, and other constituents found in various essential oils, and it is to these reactions that I wish to draw attention now.

The reagent mentioned above is prepared as follows:

Ten grammes of mercuric sulphate are dissolved in and made up to 100 c.c. with 25 per cent. pure sulphuric acid.

Then 2 c.c. of the substance to be examined are placed in a small phial fitted with a cork, and 5 c.c. of the reagent added. The whole is then vigorously shaken, any change in colour noted, and again examined after standing for about ten minutes. A porcelain tile will be found useful when only small quantities of an oil can be obtained. The sensitiveness of the colour reaction is not in any way impaired by the use of such small quantities. One drop of the oil is placed on the tile and 3 to 4 drops of the reagent added, and well stirred with a glass rod.

Twenty-five per cent. sulphuric acid itself gives no reaction with the substances I have examined.

The following reactions are characteristic:

Citral.—Bright red colour forms on shaking, which rapidly disappears, and at the same time a whitish compound is formed, which floats on top of aqueous portion.

Citronellal.—A bright yellow colour forms on shaking. The compound formed retains the yellow colour for some time.

Limonene.—A very faint flesh colour at first forms, but almost instantly disappears, leaving a white compound.

Linalyl Acetate.—A brilliant violet colour, which remains permanent, increasing slightly in depth.

Linalol.—Quickly gives a deep violet colour.

Carophyllene.—A yellowish compound, but not any violet colour.

Eugenol.—A slight violet colour on standing for some time.

Cinnamic Aldehyde.—No reaction.

Terpineol.—Flesh colour and precipitate.

Formic Aldehyde.—No reaction.

Acetic Aldehyde.—No reaction.

Benzaldehyde.—No reaction.

Anisic Aldehyde.—No reaction.

The following essential oils have also been examined:

Oil of Cassia.—Yellowish compound formed, floating on the oil. No reduction on shaking.

Oil of Cinnamon.—Brown compound formed and slight violet-coloured aqueous portion. On standing, the whole becomes a black, solid mass.

Oil of Cloves.—On shaking and standing for some time, a violet colour is formed in the aqueous portion, and increases on standing.

Discussion.

The President having invited discussion,

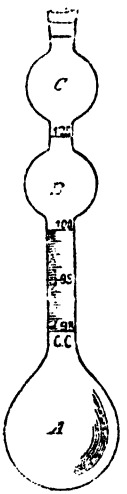
Mr. Chapman said that it would be interesting to know something of the nature of the compounds which the author had observed to be formed with the mercuric sulphate. A great deal of interest attached to many colour reactions, and more especially to those which afforded means of readily identifying some of the constituents of essential oils. But their general usefulness was to a great extent limited by the fact that they were, more than any other reactions, subject to the disturbing influence of impurities. The compounds existing in essential oils were very often extremely difficult to separate in a state of purity, when working with small quantities of material, and consequently it was possible that these colour reactions might on that account have a somewhat limited application.

ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

FOODS AND DRUGS ANALYSIS.

Examination of Brown and Taylor's Official Method of identifying Butter. J. A. Hummel. (*Journ. Amer. Chem. Soc.*, 1900, xxii., 327.)—In the manufacture of renovated butter, the butter-fat is melted and then cooled rather rapidly in a stream of cold water. This melting and rapid cooling induces a semi-crystallization of the fat. Samples of genuine and renovated butter were examined with a polarizing microscope magnifying 120 diameters having a selenite plate below the slide. Normal butters gave a uniformly blue field. Renovated butters gave a blue field mottled with yellow. A. M.

New Method for the Determination of Essential Oils in Drugs and Spices. Neumann-Wender and G. Gregor. (*Oest. Chem. Zeit.*, 1900, iii., 233-235.)—The method depends upon the fact that petroleum ether, of specific gravity 0·640 to 0·670, does not mix with alcohol of 40 to 60 per cent. strength (Hefelmann, *Pharm. Centralhalle*, 1896, 683), whereas essential oils can be readily shaken out of alcoholic solutions by means of the petroleum ether.



Ten to 20 grammes of the coarsely-powdered substance are heated for six hours with 70 c.c. of 96 per cent. alcohol. The residue is again treated for six hours with 25 c.c. more of the alcohol. The extracts are united and made up to 100 c.c.; 50 c.c. of this are then taken and distilled with steam until the distillate is no longer milky. A distillate of 95 c.c. is generally sufficient. This is made up to 100 c.c., 50 c.c. of it are introduced into the apparatus shown in the illustration, and are made up to the 100 c.c. mark with water acidified with a few drops of sulphuric acid; 25 c.c. of petroleum spirit are then introduced, and the apparatus is well shaken and allowed to stand some hours. The decrease in volume of the alcoholic solution gives the quantity of ethereal oil. A. M.

The Gluten Constituents of Wheat and Flour and their Relation to Bread-making Qualities. H. A. Guess. (*Journ. Amer. Chem. Soc.*, 1900, xxii., 263.)—The analytical work was performed as follows:

(*a*) Five grammes of the finely-ground wheat meal or flour were placed in a 250 c.c. flask, which was then filled to the mark with 1 per cent. salt solution, shaken at intervals for an hour, and allowed to settle for two hours. The liquid was then decanted through a filter, 100 c.c. of it taken, and the proteids precipitated with a few c.c. of 10 per cent. solution of phosphotungstic acid. The precipitate was allowed to settle, 50 c.c. of the clear filtrate were evaporated with sulphuric acid, and the amide nitrogen determined.

(*b*) One gramme of sample was put into a 500 c.c. flask with 100 c.c. alcohol (specific gravity 0·90), shaken thoroughly, and heated with occasional shaking for one hour just below the boiling-point. It was then allowed to settle for one hour, and the clear liquid decanted off. Twenty-five c.c. fresh hot alcohol were added, allowed to settle for twenty minutes, decanted, and the operation repeated three times. In the extract the alcohol was distilled off, and the nitrogen in the residue determined, the amide nitrogen (*a*) subtracted, and the remainder calculated as gliadin (N × 5·7).

(*c*) To the residue from the alcohol extraction, after cooling, 250 c.c. of 1 per cent. salt solution were added. It was allowed to settle for one hour, and decanted through a filter. Two hundred and fifty c.c. fresh salt solution were added, shaken at intervals for one hour, allowed to settle two hours, and decanted through the same filter. The filter and contents were added to the residue in the flask, and the nitrogen determined and calculated to glutenin.

The author found that the quality of the gluten depends upon the ratio in which its two constituents are present. The higher the ratio of gliadin to glutenin, the more elastic is the gluten, and consequently the better is the result of a baking test.

The author publishes the result of the analysis of eighty wheats from the Canadian North-West. The gliadin in these varies from 3·65 to 9·21 per cent. The glutenin varies from 3·30 to 5·64, and the ratio from 0·70 to 2·90. A number of flours were also examined. The results show that, whilst the gliadin remains fairly constant, the glutenin increases as one goes through the different grades in descending scale. Thus, in Keewatin flour the gliadin was always about 8 per cent., but the glutenin varied from about 2 per cent. in the patent grade to over 5 per cent. in the Glenwood grade.

As an aid in grading wheats and flours for commercial purposes, the author proposes a "composite factor," which is the product obtained by multiplying the percentage of gluten (*i.e.*, gliadin + glutenin) by the ratio of gliadin to glutenin. In the case of the Keewatin flours, the factor comes out at 37 to 58 for patent grades, and 15 to 21 for Glenwood grade, the intermediate grades giving intermediate figures.

A. M.

Detection of Cherry Syrup in Raspberry Syrup. O. Langkopf. (*Pharm. C. H.*, 1900, xl., 421; through *Chem. Zeit. Rep.*, 1900, 219.)—The author's process depends on the presence of hydrocyanic acid in cherry syrup. If one drop of an alcoholic solution of guaiacum is mixed with a little 1 : 10,000 copper sulphate solution, a milky liquid is produced which turns blue with an exceedingly minute trace of HCN. From 50 or 100 c.c. of the syrup to be examined 2 c.c. are distilled, and the distillate collected in some of the reagent, when 5 per cent. of cherry syrup in raspberry causes the appearance of the blue tint.

F. H. L.

The Examination of Gum Acacia. R. G. Shoults. (*Amer. Journ. Pharm.*, 1900, lxxii., 267-270.)—According to the United States Pharmacopœia (1890), pure gum acacia does not reduce Fehling's solution. The author, however, shows that

this statement is not correct. He found that powdered samples which caused only a slight reduction at the temperature of the water-bath, yielded much heavier precipitates when the supernatant liquid was filtered and again boiled; and that samples which yielded no deposit at first gave a marked reduction after being dried at 130° C. and then boiled with the Fehling's solution. In fact, this phenomenon was observed after simple powdering of the gum without drying.

He considers that if dextrin were used as an adulterant, a white sample would be liable to contain unconverted starch, which would be detected by means of iodine. A high specific rotatory power, in combination with the detection of starch, would certainly indicate dextrin. On the other hand, a dextrin free from starch would be more or less yellow in colour.

For the calculation of the amount of dextrin (specific rotatory power = 198) the author subtracts the specific rotatory power of pure acacia (18) from the observed specific rotatory power of the sample, and divides the results by the increase in the rotatory power produced by an addition of 1 per cent. of dextrin (viz., 1·8). Thus, if a specimen of powdered acacia had a specific rotatory power of 126, this would correspond to $126 - 18 = 108$; $108 \div 1{\cdot}8 = 60$ per cent. of dextrin. C. A. M.

Euquinine. Zimmer and Co. (*Special pamphlet;* through *Chem. Zeit. Rep.*, 1900, 159.)—Euquinine, or euchinine ("quininæ ethyl-carbonas"), is quinine ethylic carbonate. It forms hard, white, needle-shaped crystals, melting at 95° C., sparingly soluble in water, easily in alcohol and ether. It has a basic reaction, and yields crystalline salts. Like quinine, euquinine shows a blue fluorescence in sulphuric acid solution, gives the thalleioquin reaction, but not the Herapathite (iodo-sulphate) test. It possesses only a faint bitter taste, and is more permanent when exposed to light than the compounds of quinine itself. F. H. L.

Determination of Bismuth in Organic Preparations. C. Gaebler. (*Pharm. Zeit.*, 1900, xlv., 567; through *Chem. Zeit. Rep.*, 1900, 219.)—In "Airol" (bismuth oxyiodogallate) the metal cannot be determined by incineration, as the bismuth volatilizes. By precipitation as oxalate, however, useful results may be obtained. "Orphol" (the β-naphtholate) can be safely ignited; according to the formula, it should contain 80·3 per cent. of Bi_2O_3. With bismuth salicylate, incineration, extraction of the residue with nitric acid, and ignition is quite accurate. "Xeroform" (bismuth tribromophenol) can neither be analysed by ignition nor by the oxalate process; it should be treated with strong nitric acid, thrown down with ammonium carbonate, and the precipitate converted into Bi_2O_3. F. H. L.

ORGANIC ANALYSIS.

Influence of Temperature on the Specific Rotatory Power of Saccharose. **F. G. Wiechmann.** (Special pamphlet, 1900.)—In this paper the author quotes and discusses the discordant statements which have been made by different observers as to the effect of varying temperature upon the opticity of cane-sugar solutions, records numerous results obtained by himself and his assistants, and finally comes to the conclusion that, under the practical working conditions existing in technical sugar analysis, the specific rotatory power of saccharose may be regarded as constant.

F. H. L.

The Halphen Reaction for Cotton Oil. **P. N. Raikow.** (*Chem. Zeit.*, 1900, xxiv., 562, 583.)—This article contains an account of some incomplete investigations of the mechanism of the Halphen reaction, as well as sundry speculations upon the true cause of the colour. Tortelli and Ruggeri have proved that the active substance in the Becchi test must be of the nature of an unsaturated acid; Raikow has demonstrated that the active substances in the Becchi and Halphen tests are so much alike as regards their removal from or destruction in the oil, that the latter may well be an unsaturated acid also, while his previous work suggested that the two substances might even be one and the same. When, however, 10 c.c. of cotton oil are shaken in a stoppered tube for two hours with dilute sulphuric acid, and increasing quantities of potassium permanganate are added by degrees, if the residual oil is collected by means of ether, filtered, and the solvent removed, a point is soon reached (5 grammes of permanganate) where the product gives a normal Becchi test but fails to yield the Halphen reaction entirely. This shows that the active substances are not the same, and also that the body which produces the Halphen colour is far more easily attacked by nascent oxygen than the substance responsible for the Becchi colour.

Raikow has mentioned (ANALYST, this vol., 106) that a Halphen red appears when the usual mixture of cotton oil, carbon bisulphide, sulphur, and amylic alcohol is simply exposed to sunlight. If such a mixture, after becoming red, is left in the light, it gradually and slowly loses its colour, turning yellow almost as at first, similar changes occurring in a sealed tube. If the original mixture, before becoming red, is kept in the dark in an open tube, it does not turn red even in eight months; and if the red insolated mixture is placed in the dark before it loses its colour it remains red indefinitely. If the red colour is produced by heating on the water-bath, further heating does not destroy it. A mixture which has been insolated till it has become yellow again may be heated on the water-bath without effect; and if more amylic alcohol, carbon bisulphide, and sulphur are added, further heating only produces an insignificant tint, such as is exhibited by olive oil containing 0·5 per cent. of cotton oil. These experiments indicate that the red body is neither produced nor destroyed in the dark; in sunlight it is first produced, and then decomposed into something different from the original active substance of the oil. Three closed tubes were exposed to south light for seven winter months, (*a*) containing cotton oil alone, (*b*) cotton oil and carbon bisulphide without free sulphur, and (*c*) cotton oil and flowers of sulphur. Tubes *b* and *c* were then tested with amylic alcohol, bisulphide,

and sulphur; they gave no reaction; tube *a* gave both Halphen and Becchi tests normally. Light alone, therefore, does not render cotton oil indifferent to the Halphen test, but in presence of sulphur and carbon bisulphide the active substance is destroyed. Part of the contents of tube *b* were freed from bisulphide by means of a current of warm air; the residual oil gave a reaction of sulphur with phloroglucinol and vanillin. There is no evidence to show whether this sulphur existed in the free state, as a compound of bisulphide with some constituent of the oil, or as a compound of sulphur and cotton oil; the latter supposition is the more likely, explaining well why carbon bisulphide ultimately makes cotton oil fail to respond to the Halphen test. In sunlight free sulphur renders the oil inactive more rapidly than bisulphide; presence of amylic alcohol hastens either process notably. The Halphen reaction is always accompanied by the evolution of sulphuretted hydrogen, and this evolution is more marked in the presence of amylic alcohol than in its absence. This liberation of gas, indeed, may be employed as a check on the intensity of the Halphen red. When two similar tubes are charged with equal volumes of the Halphen mixture, if the colours in both develop equally, moist lead papers placed over the mouths of the tubes show identical brownish-black tints; if the reds are different, parallel differences are to be seen in the stains of lead sulphide.

All the foregoing observations give the active substance the character of an unsaturated fatty acid. Yet 95·8 per cent. of the acids of cotton oil are solid, saturated bodies, and, according to Hazura and Grüssner, the 4·2 per cent. of liquid acids are composed of linolic and oleic, which occur in nearly all vegetable oils, and which do not give the Halphen reaction. Therefore it may be asserted that the red colour depends on some unsaturated acid not hitherto isolated, which is only present in a very small proportion.

Finally, Raikow calls attention to Barbaglia's work upon valeraldehyde and benzaldehyde when heated with sulphur, and to Gattermann's statement that the thiocarbonyl group is a chromophore, whence it seems not improbable that the action of sulphur upon the unsaturated acid of cotton oil may be either to convert a CH_2 group into CHS, or a CH group into CS. When, accordingly, the Halphen colour is produced by heat alone, the thiocarbonyl group suffers no further change; but if the reaction proceeds in sunlight, or if the heated red mixture is insolated, a second stage of the reaction occurs in which the thioaldehyde or thioketone is altered or polymerized, liberating H_2S, and losing its red colour [*cf.* the reactions of benzaldehyde, *Journ. Chem. Soc.*, Abs., 1891, p. 1049, and of valeraldehyde, *loc. cit.*, 1885, p. 136]. Thus the thiocarbonyl group is wholly broken up, yielding a derivative indifferent to sulphur, and this explains why cotton oil which has lost its Halphen red will not give the test a second time. Cotton oil never yields so strong a red in sunlight as on the water-bath, because in the former case production of the CS group is accompanied by its almost simultaneous destruction. It is rather curious, in spite of the extremely small quantity of the active substance present in the oil, that so large an amount of permanganate should be required to decompose it. This may be explained by the hypothesis that the active body is less susceptible of oxidation than at least part of the other unsaturated acids, whence it follows that the said active substance must be no less nearly saturated than oleic and linolic acids. F. H. L.

Maize Oil (Corn Oil). H. T. Vulte and H. W. Gibson. (*Journ. Amer. Chem. Soc.*, 1900, xxii., 453.)—Three samples were examined. No. I. was six or seven years old. No. II. was a sample freshly prepared by hydraulic pressure, and was of undoubted purity. No. III. was a product of the mash of distilleries:

	I.	II.	III.
Specific gravity at 15·5° C.	0·9213	0·9213	0·9255
,, ,, 100° C.	0·8716	0·8711	0·8756
Viscosity (water = 1) 20° C.	9·79	10·57	—
,, (rape oil = 100) 20° C.	70·42	73·89	—
Index of refraction at 15° C.	1·4767	1·4766	—
,, ,, 20° C.	1·4761	—	1·4765
Ash per cent.	0·065	—	0·0655
Acid value	3·70	2·25	20·65
Iodine absorption	119·7	118·6	113·3
Saponification value	192·7	192·6	191·8
Hehner value	92·8	92·2	88·2
Reichert value	4·3	4·2	9·9
Acetyl value	11·1	11·5	—
Glycerol per cent.	10·55	10·35	—
Unsaponifiable matter per cent.	1·39	1·43	—
Maumené—rise in temperature	75° C.	74° C.	—
,, —specific ,, ...	179	176	—
Bromine thermal value	21·9° C.	21·8° C.	—
Valenta's test	74° C.	80° C.	65° C.
Livache test—per cent. gain	5·97 in seven days	5·19 in ten days	—

The fatty acids gave the following results: Specific gravity at 100° C., 0·8529; melting-point, 22·4° C.; iodine absorption, 121·0; saponification value, 199·1; bromine thermal value, 21·6° C.

The iodine values were determined by Hübl's method, using an excess of 100 per cent. and allowing to stand twenty-four hours. A. M.

The Composition and Analysis of Artificial Turpentine. G. Fabris. (*Annali del Laboratorio delle Gabelle*, 1900, iv., 143-150.)—The substitutes for turpentine which have been on the market for some time consist, according to the author, of mixtures of rosin oil, colophony, and oil of turpentine. Their colour and consistency depend on the relative proportion of these constituents, and they usually have a mingled odour of colophony and rosin oil.

Six commercial samples examined by the author were all completely soluble in 95 per cent. alcohol, and on distillation yielded volatile compounds (turpentine oil) below 250° C.

On analysis they gave the following results: Substance volatile below 250° C., 6·5 to 13 per cent.; acid value, 105 to 113·8; saponification value 113·6 to 119·2; difference between saponification and acid values 5·3 to 9·2.

From these figures the author calculated approximately the composition of the samples from the data that the rosin oil used boiled above 300° C. and had no appreciable acidity; that oil of turpentine boils at 151 to 170° C., and is not acid when

pure; and that colophony commonly has an acid value of 161 to 170. They were calculated to have the following composition: Turpentine oil (substance volatile below 250° C.), 6·0 to 13 per cent.; colophony (from acid value) 65·2 to 67·9 per cent.; and rosin oil (by difference) 19·1 to 28·8 per cent.

In addition to the distinct difference in appearance and odour, natural turpentine (French or American) contains more than 15 per cent. of constituents volatile below 200° C., whilst the highest amount found by the author was 13 per cent. in one sample, the others yielding only 6 or 7 per cent. The artificial products also contain rosin oil, which is readily recognisable.

Larch or Venetian turpentine might possibly be confounded with the artificial preparations, but it invariably contains more than 15 per cent. of oil of turpentine. According to Dieterich, the acid value of Venetian turpentine varies from 65 to 75, and the saponification value from 110 to 125. In three commercial samples the author found 20 to 25 per cent. of oil of turpentine and acid values of 90·1, 90·2, and 96·8. The difference between the acid and saponification values is thus much greater than in the artificial products.

In order to detect rosin oil, 5 grammes of the turpentine are dissolved in 20 c.c. of 95 per cent. alcohol, a few drops of phenol-phthalein added, and the solution rendered just alkaline with potassium hydroxide. In the case of artificial turpentine the liquid becomes turbid, and, on standing, oily drops of rosin oil separate out, whereas Venetian turpentine tested in the same way gives a perfectly clear solution. C. A. M.

Oil of Bitter Orange. **G. Fabris.** (*Annali del Laboratorio delle Gabelle*, 1900, iv., 139, 140.)—Oil of bitter orange, which is also known as *Essence d'orange bigarade*, is extracted from the peel of the bitter orange. Having only a very limited use, it is not, according to the author, easily obtained pure in commerce.

It is a yellow liquid, with a bitter aromatic taste, and an odour of orange rather more delicate than oil of sweet orange. Its rotatory power varies within far wider limits than that of the sweet oil ($+92°$ to $98°$), but it does not differ much from the latter in its other properties. Two pure specimens examined by the author gave the following results: Specific gravity at 15° C., 0·853 and 0·852; and rotation at 20° C., 92°·40′ and 92°·0′. On account of the greater variation in the rotatory power, this oil can be more easily adulterated than oil of sweet orange. But the author considers that the rotation considered in conjunction with a fractional distillation should be sufficient to detect the more common and grosser forms of adulteration. C. A. M

Detection of Salicylic Acid in Presence of Citric Acid. **A. Klett.** (*Pharm. C. H.*, 1900, xli., 452; through *Chem. Zeit. Rep.*, 1900, 238.)—The following test, due to Jorissen, will indicate the presence of salicylic acid in lemon-juice by giving a blood-red colour: 10 c.c. of the liquid are mixed with 4 drops of a 10 per cent. solution of potassium or sodium nitrite, 4 drops of acetic acid, and 1 drop of 10 per cent. copper sulphate, the whole being heated to the boiling point. (*Cf.* this vol., p. 238.) F. H. L.

Detection of Salicylic Acid and Vegetable Albumin by Means of Millon's Reagent. C. J. Lintner. (*Zeits. angew. Chem.*, 1900, 707.)—When prepared in the usual manner, by dissolving mercury in nitric acid, Millon's reagent tends to give somewhat uncertain results; the author therefore prefers to use solutions of mercuric nitrate and sodium nitrite, keeping them separately and adding them in succession to the substance under examination. For the detection of salicylic acid, the sample should be boiled for two minutes with a few drops of 10 per cent. mercuric nitrate, then 2 or 3 drops of dilute sulphuric acid and a very small quantity of 1 per cent. sodium nitrite solution, avoiding excess, should be added. The red colour appears usually with the first drops of nitrite, and generally becomes more intense as the mixture cools. It will remain unaltered for a day. Dilute nitric acid can be employed to acidify the liquid, but it gives a yellow instead of a blue shade of red to the reaction, and thus diminishes the delicacy of the test. This limit of delicacy, both with Millon's reagent and the above modification, is at about 1 part of salicylic acid per 500,000; the modified test yields rather brighter and more permanent tints.

To detect vegetable albumin in seeds, etc., a combined reagent should be prepared of the mercuric nitrate solution, 5 vols.; the sodium nitrite, 1 vol.; and dilute sulphuric or nitric acid, 1 vol. If, for example, longitudinal sections of barley grains are placed in this liquid, the cut surfaces gradually turn red-violet, which renders the glutinous layer and the embryo very conspicuous; the husk becomes a dark brownish-red. F. H. L.

Detection of Indican in Pathological Urine. A. Klett. (*Chem. Zeit.*, 1900, xxiv., 690.)—The ordinary calcium hypochlorite test for indican is very apt to fail, especially if the reagent be added in excess; and it is better to use ammonium persulphate, which gives a blue coloration unaffected by excess. To 10 c.c. of urine are added 5 c.c. of 25 per cent. hydrochloric acid, some persulphate, and then chloroform. Klett calls attention to Strzyowski's test for albumin and bile pigments (ANALYST, 1899, xxiv., 49), and observes that ammonium persulphate is a most serviceable reagent in the examination of urine. F. H. L.

The Relation of the Reducing Power of Normal Urine to the Amount of certain Nitrogen Compounds Present. J. H. Long. (*Journ. Amer. Chem. Soc.*, 1900, xxii., 309.)—Normal urine contains only a small proportion of sugar (see Allen, ANALYST, 1894, 178.) Among the other substances present which have the power of reducing copper, the most important are uric acid and creatinine. The author has determined their reducing power, and has estimated the amount present in a number of normal urines. For this work a sensitive copper solution was required. It was found that the oxidizing power increases with the amount of caustic soda and ammonia present. The solution adopted had the following composition: Copper sulphate, 8·166 grammes; sodium hydroxide (100 per cent.), 15 grammes; glycerol, 25 c.c.; ammonia (specific gravity 0·9), 350 c.c.; water to make 1 litre. One c.c. of this oxidizes 1 milligramme of sugar in 0·2 per cent. solution; 50 c.c. of the solution are taken, diluted to 100 c.c.; solid paraffin is added, which when melted covers the

surface. The liquid is boiled gently and the solution under examination is added slowly; 1 molecule of dextrose = 5·88 molecules of CuO.

It was found that 1 molecule of creatinine, $C_4H_7N_3O$, reduces 2 molecules, CuO, and 1 molecule of uric acid, $C_5H_4N_4O_3$, almost 3.

The following table gives the results of the examination of 24 samples of healthy urine, obtained mostly from young men:

	Minimum.	Maximum.	Mean.
(*a*) Excretion in twenty-four hours	765 c.c.	1920 c.c.	1167 c.c.
(*b*) Specific gravity at 20° C.	1·018	1·035	1·025
(*c*) NH_3, milligrammes per litre	227·7	1001·0	620·0
(*d*) Uric acid, milligrammes per litre ...	423·7	1020·0	658·7
(*e*) Creatinine ,, ,, ...	653	1930	1392
(*f*) Urea, grammes per litre	14·83	37·27	24·37
(*g*) Total reducing power, grammes CuO per litre	2·245	8·738	6·204
(*h*) Reducing power of uric acid	0·592	1·381	0·935
(*k*) ,, ,, creatinine	0·920	3·133	1·961
(*l*) Sum of *h* and *k*	1·897	4·023	2·896
(*m*) Ratio of *l* to *g*	0·328	0·631	0·466

The reducing power, which remains unaccounted for, is probably due principally to saccharine matter. Calculation shows that the quantity of the latter present amounts in the mean to about 1·4 grammes dextrose per litre. The value (*m*) was found to be highest for men of strong physique with a diet containing meat. A man consuming a diet largely meat gave values 0·63 and 0·60. The same individual some weeks later, after a change to bread and vegetables largely, gave a value 0·3.

The urea figures given above were determined by the Knop-Hüfner process. Higher results were naturally obtained by the Liebig process. The ratio of urea to uric acid is 36·9. A. M.

Artificial Silks. C. Hassac. (*Oest. Chem. Zeit.*, 1900, 235-237, 267-269, 297-299.)—The materials examined were:

1. Chardonnet silk (made from collodion) from Près de Vaux, near Besançon.
2. ,, ,, from Fismes, in North France.
3. ,, ,, from Walston, in England.
4. Silk made from collodion by Lehner's process at Glattbrugg, near Zurich.
5. Cellulose silk made with ammoniacal copper oxide, by Dr. Pauly's process, at Oberbruch, near Aix-la-Chapelle.
6. Gelatin silk.

Nos. 1, 2, and 4 are very similar in *appearance*. They excel real silk in lustre, but they are stiffer and have not the characteristic feel of the real material. No. 3 feels rough and appears hairy. It reminds one of mohair rather than of silk. No. 5 resembles Nos. 1 and 2 in appearance, but its lustre is even better, and when made up it has also the characteristic feel of real silk.

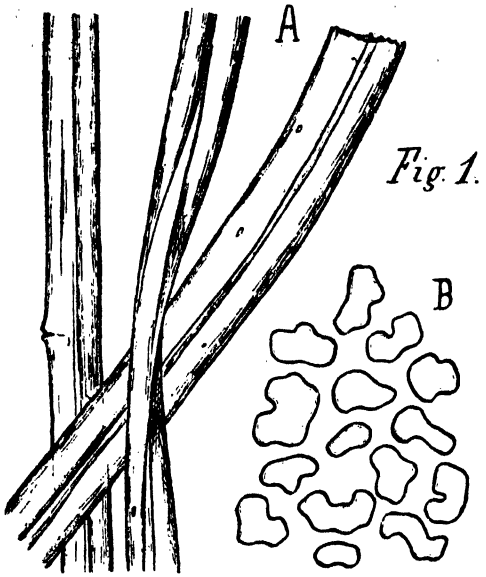


Fig. 1.

CHARDONNET SILK (COLLODION) FROM PRÈS DE VAUX.
B, Cross-section. (×150.)

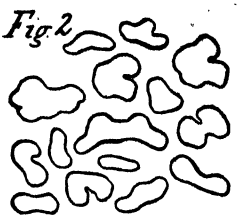


Fig. 2

CROSS-SECTION OF CHARDONNET SILK FROM WALSTON. (×150.)



Fig. 3

CROSS-SECTION OF CHARDONNET SILK FROM FISMES. (×150.)

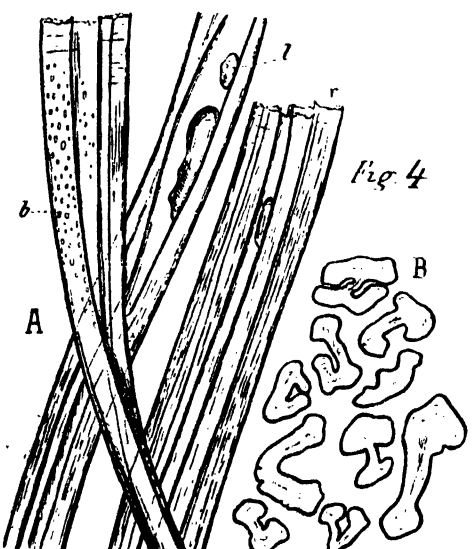


Fig. 4

LEHNER'S COLLODION SILK.
b, Minute air-bubbles in the fibre; *l*, large bubbles; *r*, fracture. B, Cross-section. (× 150.)

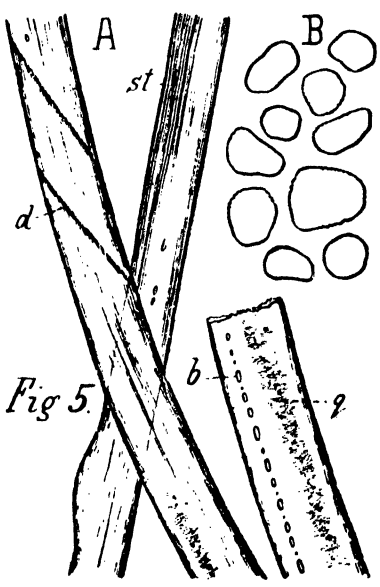


Fig 5.

PAULY'S CELLULOSE SILK.
d, Mark where two fibres have been pressed together; *st*, fine grooves; *b*, air-bubbles; *q*, cross-lines inside fibres. B, Cross-section. (×150.)

Under the *microscope* the Chardonnet silks much resemble one another, except that the Fismes product is grooved more. They can, however, be distinguished by their cross-sections (see Figs. 1, 2, 3). Lehner silk (Fig. 4) is characterized by deep grooves running lengthwise along the fibres and by small air-bubbles. Cellulose silk (Fig. 5) is regular in cross-section. On the surface are fine grooves running lengthwise (*st*), and in the centre of the fibres are fine lines running across (*q*). Gelatin silk (Fig. 6) is almost circular in section and free from grooves or bubbles. The fracture is even and the thickness constant.

The figures represent the materials swollen by immersion in water and magnified 150 diameters.

In *polarized light* all the artificial silks show double refraction, except gelatin silk, which is singly refracting. In this they resemble natural silk. In No. 5 the interference colours are even over considerable lengths of the fibres, whereas in the collodion silks great play of colours is observed in consequence of the variations in thickness.

Behaviour with Reagents. — The collodion silks, Nos. 1 to 4, always contain a small amount of nitro-compound, and consequently give a fine blue with a solution of *diphenylamine* in sulphuric acid.

When immersed in *water*, all artificial silks swell. If placed in absolute alcohol or glycerin, the fibres contract again.

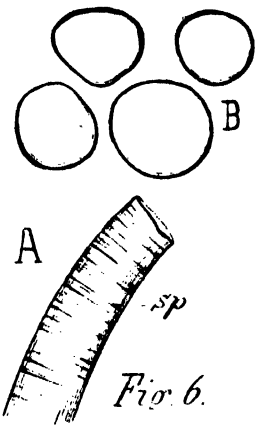


Fig. 6.

GELATIN SILK BY PROFESSOR HAMMEL'S PROCESS.

sp, Splits in a bent fibre. B, Cross-section. (×150.)

In *concentrated sulphuric acid* the collodion silks swell rapidly and dissolve. Cellulose silk becomes gradually thinner and dissolves away. Gelatin silk only dissolves on strongly heating.

Concentrated hydrochloric acid has not much action on artificial silks in the cold. When warmed it dissolves gelatin silk rapidly.

Acetic acid only causes a slight swelling, except in the case of gelatin silk, which on boiling dissolves almost completely.

Half-saturated *chromic acid* solution in the cold rapidly dissolves all artificial silks. Real silk takes longer. Cotton, flax, and other vegetable fibres are not dissolved.

Forty per cent. potash lye dissolves gelatin silk rapidly. Collodion and cellulose silks swell, but do not dissolve, even on boiling; but the liquid assumes a yellow colour. Real silk (white) is dissolved on boiling, but does not colour the liquid.

Schweizer's reagent (ammoniacal copper oxide) causes the collodion silks to swell rapidly and then dissolve. Cellulose silk swells less rapidly. Gelatin silk does not dissolve, but is coloured bright violet.

Alkaline copper-glycerin solution is a very useful reagent. Real silk dissolves immediately on warming to 80°. Tussah silk dissolves on boiling about a minute, as also does gelatin silk. The other artificial silks are in no way affected.

Solution of *iodine and potassium iodide* colours artificial silks an intense red or

reddish-brown. When washed with water the colour disappears, and collodion silks show a transient grayish-blue. Cellulose silk does not show this blue coloration.

Solution of *iodine and dilute sulphuric acid* (v. Höhmel's paper reagent) colours real silk yellow, gelatin silk yellowish-brown to reddish-brown, collodion silks deep blue with a shade of violet, cellulose silk pure blue.

Solution of *zinc chloride and iodine* colours collodion silks bluish-violet, cellulose silk grayish-blue to grayish-violet; gelatin silk, like real silk, becomes yellow and disintegrates.

As regards *inflammability*, the artificial silks, with the exception of gelatin silk, behave like cotton. Gelatin silk behaves in this respect like real silk.

In the following table the first column shows the moisture in the air-dry material. The second column the moisture after standing twenty-four hours in an atmosphere saturated with water vapour. The third shows the specific gravity, determined by weighing in benzene, air-bubbles being removed by manipulation under the receiver of a vacuum pump. The fourth column shows the number of elementary fibres which go to 1 square millimetre after soaking in water. This was determined by measuring the sectional area under the microscope. The fifth column shows the number of fibres in the air-dry state which go to 1 square millimetre. This was calculated from the previous figures and the known coefficients of swelling, as satisfactory sections could not be cut dry. The tensile strengths were measured on the instrument of Teclu (*Centralorgan f. Wkd. u. Technlg.*, i., 200).

	Moisture		Specific Gravity.	No. of Fibres to 1 sq. mm.		Tensile Strength. Kg. per sq. mm.		Extension. Per cent.
	Air-dry. Per cent.	Saturated. Per cent.		Wet.	Dry.	Wet.	Dry.	Dry.
Real silk ...	8·71	20·11	1·36	9,710	9,710	37·0	37·0	21·6
1. Chardonnet	11·11	27·46	1·52	640	1,135	2·2	12·0	8·0
2. Fismes ...	10·92	27·12	1·52	370	656	1·6	7·8	11·6
3. Walston ...	11·32	28·94	1·53	683	1,620	1·0	22·3	7·9
4. Lehner ...	10·45	26·45	1·51	413	1,180	1·5	16·9	7·5
5. Cellulose ...	9·20	23·08	1·50	742	1,550	3·2	19·1	12·5
6. Gelatin ...	13·98	45·56	1·37	265	945	nil	6·6	3·8

It will be seen that the cellulose silk made by Dr. Pauly's process is the most satisfactory. A. M.

INORGANIC ANALYSIS.

Titration of Mercury Salts with Thiosulphate. **J. J. Norton.** (*Zeits. anorg. Chem.*, 1900, xxiv., 411.)—Sherer having stated (*cf.* Sutton's "Volumetric Analysis") that mercurous nitrate, mercuric nitrate, and mercuric chloride can be accurately determined by titration with standard thiosulphate, the present article is a study of the processes suggested. Norton finds that neither mercurous nor mercuric nitrate can be analysed by Sherer's method; but that mercuric chloride is amenable to the process if the following directions are followed closely: The solution, containing up to about 0·2 gramme of metallic mercury, is brought into a 1-litre flask, diluted to 100 c.c., heated to 60° C., and a $\frac{N}{20}$ solution of thiosulphate is run in till the precipitate, which is at first white, becomes brownish. The liquid is then diluted with

cold water, some asbestos fibres added to assist filtration, and the whole filtered through asbestos with the aid of the pump. After thorough washing, the filtrate is made up to a definite volume, treated with 3 grammes of potassium iodide, and the excess of thiosulphate is titrated with iodine and starch. The process should not occupy more than fifteen minutes. Addition of hydrochloric acid is not necessary.

F. H. L.

Test for Tin. **A. Rogers** (*Journ. Amer. Chem. Soc.*, 1900, xxii., 220) and **J. P. Longstaff** (*ibid.*, 450).—The separation of the tin is conducted in the usual way with zinc, and the black flakes dissolved in hydrochloric acid. To a few drops of this solution a little water is added, and then ammonium molybdate. A blue colour shows the presence of tin.

Longstaff points out that he published the method last year in the *Chem. News* (lxxix., 282). The zinc which is used for reduction must be quite free from tin. With dilute solutions the molybdate should be added at once, before the tin can become oxidized. One part of stannous chloride can thus be readily detected in 100,000 parts of solution. By using the special precautions described in the *Chem. News*, it is possible to detect 1 in 1,500,000.

A. M.

Valuation of Zinc Dust. **A. Fraenkel.** (*Mitth. Techn. Gew. Mus. Wien*, 1900, x., 161; through *Chem. Zeit. Rep.*, 1900, 219.)—One gramme of the sample is weighed into a dry stoppered 200 c.c. flask, mixed with 100 c.c. of potassium bichromate solution (30 grammes per litre) and 10 c.c. of 1 : 3 sulphuric acid, and agitated for five minutes. Another 10 c.c. of acid are then added, and the shaking continued for ten or fifteen minutes, when everything, except a small earthy residue, should be dissolved. The liquid is diluted to 500 c.c., and in 50 c.c. thereof the excess of bichromate is estimated by introducing 10 c.c. of 10 per cent. potassium iodide and 5 c.c. of sulphuric acid, titrating the liberated iodine with decinormal thiosulphate.

F. H. L.

Estimation of Cobalt in New Caledonian Ores. **T. Moore.** (*Chem. News*, 1900, lxxxii., 66.)—These ores consist mainly of the hydroxides of manganese, iron, aluminium, cobalt, and nickel, with small quantities of calcium, magnesium, zinc, and lithium; occasionally also barium and copper. The proportion of cobalt may reach 8 per cent., averaging 5 per cent., and the ores are sold simply upon the amount of that metal. To estimate the cobalt, 2·5 grammes are dissolved in strong hydrochloric acid, and evaporated to a syrup; the soluble matter is taken up in water, and the Fe and Al precipitated by the basic process. To the filtrate 20 c.c. of saturated sodium acetate and 10 c.c. of acetic acid are added, and the whole heated nearly to boiling. A current of sulphuretted hydrogen is next passed through the solution till it is almost cold, when the sulphides of cobalt, nickel, and zinc are collected, washed with H_2S-water, dried, and ignited. The ignited oxides and any sulphide adhering to the walls of the flask are dissolved in strong HCl and a few drops of HNO_3, evaporated twice to dryness with more HCl, and the chlorides dissolved in 5 or 10 c.c. of water. To remove any traces of iron which

may have escaped precipitation, this solution is treated with a cream of zinc oxide. The filtrate, diluted to about 50 c.c., is mixed with 10 or 15 c.c. of 10 per cent. hydrogen peroxide and 10 c.c. of 10 per cent. sodium hydroxide. The precipitate of cobalt sesquioxide and nickel monoxide is boiled for a minute, cooled, and treated in a stoppered bottle with excess of potassium iodide and HCl until the decomposition is complete, titrating the liberated iodine with thiosulphate ($I \times 0{\cdot}46511 = Co$). The caustic soda employed must be examined for impurities capable of liberating iodine, as samples containing nitrites are not uncommon. The examples quoted are satisfactory. F. H. L.

APPARATUS.

The Use of Floats in Burettes. Kreitling. (*Zeits. angew. Chem.*, 1900, 829.)—This is a lengthy article on the behaviour of an Erdmann float in a graduated burette, and the author's conclusions are as follows: It is advisable never to use a float in a burette, for the readings obtained with this device vary considerably at different times and with different observers. In etched burettes graduated without a float, the device should on no account be adopted, for it is liable to cause errors the magnitude of which cannot be predicted. F. H. L.

The Distillation of Ammonia in the Determination of Nitrogen. F. G. Benedict. (*Journ. Amer. Chem. Soc.*, 1900, xxii., 259.)—Ammonia is entirely removed from an alkaline liquid after boiling for quite a short time. If a condenser be used, part of the ammonia condenses in it, and requires a large amount of distillate to wash it through—not less than 150 c.c. If, on the other hand, the condenser be done away with, and the steam be led directly into a beaker containing standard acid, the latter becomes very hot, and must be cooled before titrating. Some of the acid may be lost also. The advantages of both these methods are combined in the following: A condenser is used, the tube of which dips below the surface of the acid in the receiver. After boiling for fifteen minutes, the water is gradually run out of the condenser, and steam is allowed to pass through for five minutes. By that time all the ammonia is distilled from the interior of the condenser. A. M.

REVIEW.

FOOD AND DRUGS. A Manual for Traders and others, being a Consolidation of the Sale of Food and Drugs Act, 1875, the Sale of Food and Drugs Act Amendment Act, 1879, Margarine Act, 1887, and Sale of Food and Drugs Act, 1899. By CHARLES JAMES HIGGINSON. London: Effingham Wilson, 11, Royal Exchange, 1900. Price 2s. 6d.

THIS is a useful little work, which contains, besides the text of the four statutes above cited as chief contents, those clauses of the four Acts which are concurrently in force, consolidated in such a manner that one can see at a glance in how far they modify or enlarge each other. The more important of the many High Court decisions which interpret the clauses of the Acts of Parliament are also shortly and clearly referred to. O. H.

THE ANALYST.

NOVEMBER, 1900.

PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS.

ON UNIFORMITY IN SOIL ANALYSES.

By A. D. Hall.

(Read at the Meeting, June 6, 1900.)

The following recommendations have been drawn up by a committee of the Agricultural Education Association,* with the aim of securing a certain uniformity in the conduct of soil analyses. The object the committee has in view in seeking for such uniformity is: (1) To enable analyses executed and published by any investigator to be intelligible to his fellow-workers, and therefore on occasion to be serviceable in their research; (2) to insure that the large number of soil analyses now being carried out at various institutions in this country shall be mutually comparable, and available for tabulation and general discussion; (3) to put on record a method of procedure that will serve as a guide to investigators beginning such work.

As the desirability of uniformity in methods of analysis has sometimes been called in question, the committee wishes to indicate in what respects it considers agreement should prevail, and the weight it attaches to its recommendations. In the first place, the committee would disclaim any idea of dictating methods in cases of what may be termed research; every worker in so incompletely surveyed a field as soil analysis affords must have ideas of his own for the examination of special points. The recommendations of the committee deal with those determinations about which there is a general consensus of opinion—such determinations, in fact, as would generally accompany a record of results of field experiments, etc. Secondly, the committee does not wish to formulate any recommendations as to the interpretation of soil analyses, recognising that the interpretation is still largely uncertain, and must depend to a great measure on the judgment and local knowledge of the analyst. Further, the committee's recommendations do not deal with the analytical processes or details of manipulation to be adopted in the determination

* The committee consisted of T. S. Dymond, of the Essex County Laboratories; M. J. R. Dunstan, University College, Nottingham; C. M. Luxmoore, Reading College; T. B. Wood, Cambridge University Agricultural Department; and A. D. Hall, South-Eastern Agricultural College, Wye.

of any given constituent; these are matters dependent on the skill and discretion of the individual, and for them the individual is responsible.

The committee wishes to define what may be termed the necessary "conventions" of soil analysis, for soil analysis is not like an atomic weight determination, or even the assay of a manure, in which a number having an absolute value can be sought for. The only absolute figures possible are those to be obtained by an ultimate elementary analysis of the whole soil. This, however, is never attempted: by tacit consent certain portions only are reserved as soil for analysis, and are, again, only submitted to a partial resolution, the portion reserved and the mode of attack varying with each chemist; yet the figure expressing the proportion in the soil of a constituent like potash may vary two- or three-fold, according to the method of attacking the soil that is adopted; hence the importance of agreement on such a point is manifest.

In its preliminary inquiries the committee found that no agreement existed among the chemists, either of this or other countries, on such points as the preparation of the soil for analysis, the nature of the solvent to be employed, the time of its action, etc.; and as there is no absolute criterion by which they can be decided, they must be regarded as "conventions," to be settled by considerations of expediency and mutual agreement. But as the results obtained will very largely depend upon the conventions adopted, this committee has attempted, after discussion and investigation, to so far define a common plan of action as will render the results obtained more generally serviceable, without hampering the individuality of each worker.

Recommendations.

1. *Taking Sample.*—Under ordinary conditions the sample shall be taken to a depth of 9 inches, but in case of shallow soils to such lesser depth as marks a natural line of demarcation.

The committee approves of the use of the auger as one method that may be adopted for taking samples. Several cores should be taken and mixed for analysis.

2. *Drying.*—The sample shall be air-dried for analysis. The drying may be accelerated by heating to a temperature not exceeding 40° C., but in all cases the soil should be finally left for a day or two, spread in a thin layer, and exposed to the air, at the ordinary temperature of the room.

3. *Sifting.*—A sieve with round holes, 3 millimetres in diameter, shall be used to separate the fine earth for analysis from the stones and gravel. Gentle pressure with a wooden or caoutchouc pestle, or other means, may be adopted to break up aggregates of clay and silt, but care should be taken not to crush any of the stones or lumps of chalk.

For determination of the "available constituents" the "fine earth" is used without grinding. For the other determinations, 100 grammes or more of "fine earth" is sifted through a woven sieve of 40 meshes to the inch, or a sieve with round holes of 1 millimetre in diameter. What is retained by the sieve is ground till it will pass through, and the whole mixed.

Perforated zinc, with holes $\frac{1}{8}$ inch and $\frac{1}{25}$ inch diameter, is commercially

obtainable, and forms a convenient material for the construction of the two sieves. Considerable diversity of opinion exists concerning the sieve to be used for obtaining "fine earth" for analysis; the Rothamsted chemists employ a woven sieve of ¼ inch mesh; the 3-millimetre round-holed sieve was originally suggested by Wolff; the Berlin Laboratorium für Bodenkunde employs a 2-millimetre round hole, but also analyses the finest particles separated by washing; Grandeau, and the French Comité Consultatif des Stations Agronomiques use a 1-millimetre woven sieve, and Petermann in his examination of the Belgian soils does the same. The committee suggests 3 millimetres as a convenient size; lumps of earth can be readily reduced to that diameter, whereas sifting down to 1 millimetre involves either so much crushing of lumps of earth as results in considerable fracture of the stones, or else wet sifting. The ¼ inch square mesh sieve passes in some cases a large proportion of hard stones that are further reduced with much difficulty, and yield unweathered material, not likely to be serviceable for plant nutrition for many years. The further grinding is recommended to obtain material from which a small sample for analysis can be fairly drawn.

4. *Determination of Moisture.* — The sample is dried in the steam-oven to constant weight.

5. *Determination of Loss on Ignition.*—The result shall be so expressed as not to include the carbon dioxide expelled from the carbonates nor the moisture previously determined.

6. *Determination of Nitrogen.*—Unless otherwise specified, the nitrogen shall be determined by Kjeldahl's method.

7. *Determination of Carbonate of Lime.*—The carbon dioxide evolved on treatment of the fine earth with acid is calculated as carbonate of lime.

This is regarded as a convenient measure of the "available basicity" of the soil, without discriminating between carbonates of lime and magnesia.

8. *Determination of "Total" Mineral Constituents.*—The fine earth is boiled with strong hydrochloric acid in an open flask for a short time in order that the acid may attain constant strength, and digested at the ordinary water-bath or steam oven temperature for 40 to 48 hours, the flask being loosely stoppered. In this solution the phosphoric acid and potash are determined, and other mineral constituents as desired.

The object of the committee is to obtain as thorough an extraction of the soil as is possible short of ultimate analysis. The time of the extraction is made sufficiently long to minimize errors due to variations in the actual time, the strength of the acid, or the temperature.

Unignited soil is taken since ignition effects a drastic and variable alteration of the constitution of the soil—*e.g.*, no constant proportion is found between the potash extracted from ignited and unignited soil. Hydrochloric acid is taken as the most generally effective solvent; even peaty soils are found to yield as much phosphoric acid to hydrochloric acid as to nitric acid, or aqua regia.

9. *Determination of Available Phosphoric Acid and Potash.*— Unless otherwise specified, the method described by Dr. B. Dyer (*Journ. Chem. Soc.*, 1894, Trans., p. 115) shall be followed, the quantities used being 200 grammes unground fine earth

and 20 grammes citric acid in 2,000 c.c. water, no further addition of citric acid being made.

10. *Expression of Results.*—Unless otherwise stated, results shall be expressed as percentages calculated on the fine earth in an air-dry state.

It has been thought desirable that some of the experimental results obtained by members of the committee should be set out in detail, in order to show the reasons upon which some of the recommendations are based. The experiments deal with the effect of ignition on the relative amounts of potash and phosphoric acid dissolved from soils by hydrochloric and nitric acids. In all cases HCl aq. or HNO_3 aq. means the acid of constant strength obtained by boiling the strong acid in the air at ordinary pressures.

1. Effect of ignition of soil on the amount of potash and phosphoric acid dissolved by nitric and hydrochloric acids.

(*a*) Soil digested for two days with HCl aq. (T. B. W., L. L. B., and G. D.):

		Percentages dissolved from Raw Soil.		Percentages dissolved from Ignited Soil.	
		Potash.	Phosphoric Acid.	Potash.	Phosphoric Acid.
Heavy clay	(7)	0·69	0·24	0·62	0·19
Peat	(8)	1·11	0·61	1·25	0·33

(*b*) Soil digested for two days with HNO_3 aq. (T. B. W., L. L. B., and G. D.):

		Percentages dissolved from Raw Soil.		Percentages dissolved from Ignited Soil.	
		Potash.	Phosphoric Acid.	Potash.	Phosphoric Acid.
Sticky clay	(1)	0·37	0·20	0·48	0·15
Red sand	(2)	0·39	0·27	0·54	0·22
Poor sticky soil ...	(3)	0·42	0·13	0·58	0·23
Good loam	(4)	0·41	0·26	0·34	0·15
Loam	(5)	—	0·40	—	0·26
Sand	(6)	0·14	0·12	0·19	0·10
Heavy clay	(7)	0·49	0·17	0·52	0·18
Peat	(8)	0·94	0·29	0·98	0·36

In the above experiments more potash and less phosphoric acid are generally obtained from the ignited soil, but there are several exceptions.

(*c*) Soil evaporated twice to dryness on water-bath with HCl aq., each operation taking about an hour (A. D. H.):

	Potash Extracted from Soil.	
	Raw.	Ignited.
Light sand	0·337	0·101
Rich loam	0·280	0·307

(*d*) Soil digested with HCl aq. and extract weighed (T. S. D.):

From raw soil	12·76 per cent. extracted.
From ignited soil	15·52 ,, ,,

2. Effect of continued digestion with hydrochloric acid.

(*a*) (C. M. L.):

	Insoluble Residue.	Potash.
Evaporated twice with HCl aq. ...	79·25	0·210
Digested two days with HCl aq. ...	75·70	0·305

(*b*) (A. D. H.):

Evaporated once with HCl aq., once with HCl aq. and a little HNO_3, then taken up with HCl aq. ...	0·280 per cent. K_2O.
Digested 24 hours with HCl aq. ...	0·372 ,, ,,

3. Comparative solvent action of hydrochloric and nitric acids.

(*a*) Digested on water-bath for two days; soil A, sandy pasture, poor in organic matter. Soil B, stiff clay, arable, rich in organic matter (C. M. L.):

	Phosphoric Acid extracted by		Potash extracted by	
	HCl aq.	HNO_3 aq.	HCl aq.	HNO_3 aq.
A	0·478	0·366	0·330	0·271
A	0·503	0·384	0·343	0·278
B	0·428	0·300	0·324	0·185
B	0·384	0·339	0·326	0·193

(*b*) Soils digested for two days on water-bath as before. Soil A, loam; B, London clay (pasture); C, chalky clay (T.S.D.).

	Phosphoric Acid extracted by		Potash extracted by	
	HCl aq.	HNO_3 aq.	HCl aq.	HNO_3 aq.
A	0·24	0·24	0·19	0·10
B	0·20	0·16	1·08	0·34
C	0·23	0·20	0·89	0·49

A, insoluble residue, from HCl aq. 86·48 per cent.; from HNO_3 aq. 88·50 per cent.

(*c*) Soil digested for two days on water-bath as before, peaty water-logged soil, giving on ignition a loss of 46·34 per cent. (A. D. H.):

Extracted with HCl aq. gave ... ... 0·157 per cent. phosphoric acid.
,, ,, HCl aq. and one-tenth HNO_3 aq. gave 0·159 per cent. ,, ,,
,, ,, HNO_3 aq. gave ... ... 0·165 per cent. ,, ,,

(*d*) Soils digested for two days on water-bath as before (T. B. W., L. L. B., and G. D.):

Nature of Soils.	Moisture when Air-dry.	Loss on ignition.
1. Boulder clay	3·52 ...	5·44
2. Red sandstone	2·7 ...	6·95
3. Poor sticky soil	2·67 ...	6·23
4. Good loam	3·5 ...	10·8
5. Loam	2·3 ...	8·6
6. Sand	0·7 ...	2·3
7. Heavy clay	3·62 ...	5·63
8. Peat	10·98 ...	35·52

	Potash extracted by		Phosphoric Acid extracted by	
	HCl aq.	HNO_3 aq.	HCl aq.	HNO_3 aq.
1.	0·59	0·37	0·17	0·20
2.	0·65	0·39	0·30	0·27
3.	0·63	0·42	0·21	0·13
4.	0·20	0·41	0·24	0·26
5.	0·42	0·24	0·45	0·40
6.	0·17	0·14	0·13	0·12
7.	0·69	0·49	0·24	0·17
8.	1·11	0·94	0·61	0·29

With one exception as regards potash, and two exceptions as regards phosphoric acid, hydrochloric was always a more effectual solvent than nitric acid. Some light on the superior solvent action of hydrochloric acid was obtained by estimating the silica soluble in a solution of sodium carbonate in both the original soil and the residue left after extraction by hydrochloric and nitric acids.

Soluble silica per cent. calculated on original soil:

Soil.	Raw Soil.	Ignited Soil.	Residue from HCl Extraction.	Residue from HNO_3 Extraction.
1.	0·30	0·77	11·18	7·22
4.	0·16	0·22	8·40	5·56
7.	0·32	0·26	8·94	6·94
8.	0·28	0·44	15·24	10·44

Silicates have been decomposed by the acids and silica set free, as shown by the comparatively small amounts of silica to be obtained from the raw soil; and more silicates have been broken up by the hydrochloric than by the nitric acid.

Discussion.

Dr. Dyer said that agricultural chemists must feel much indebted to the author and to the other members of the committee who had been working in connection with this matter. The subject was one which had been much in need of attention. He was very pleased to find that all idea had been discarded of laying down absolutely uniform methods for the actual determinations. It was very desirable that there should be uniform methods for dissolving from the soil the constituents to be determined, but nothing was more irksome than to have stereotyped methods for the determinations to be made in the solution obtained. Of course, in many branches of analysis the methods were necessarily arbitrary, where all that was required was to get comparable numbers under definite conditions, and not actually to determine the total percentages of given substances; but in determining the quantity of phosphoric acid in a given solution, or the quantity of nitrogen in a given material, provided that the process gave accurate results, that was all that was required. The mechanical question as to sieves was of importance, and he (Dr. Dyer) saw no reason at all, from his own experience, for disagreeing in any way with the adopted selection of admittedly arbitrary sieves. The ¼-inch sieve that had been largely used here was merely the sieve that had happened to be adopted in soil-sampling at Rothamsted; and when the same soils were under observation year after year, if the results were to be comparable, it was necessary to maintain the system of "sieving" which had been

followed at the commencement. But there seemed to be no reason at all why that should be adopted generally, and the meshes described by Mr. Hall seemed to be quite as reasonable as any others that could be suggested. With regard to the depth of the soil, in relation to sampling, it might be interesting to Mr. Wood and other agricultural chemists to know that at Rothamsted, where the depth of 9 inches had been adopted as indicating the surface soil, Sir John Lawes and Sir Henry Gilbert had arrived at the conclusion that 9 inches was probably a little too deep for average purposes. If they were beginning over again, they would sample the surface soil to a slightly less depth than that. He (Dr. Dyer) had recently had a conversation with Sir Henry Gilbert on this subject, and learnt that at Rothamsted the view now held was to the effect that a more suitable depth would be one-fifth of a metre, viz., something under 6 inches—a convenient international measure. Anything deeper than that would be regarded as subsoil. But, of course, local conditions might altogether alter this. For instance, if the ploughing had always been shallow in a certain field, the depth of ploughing would really fix what was the surface soil, as distinguished from the subsoil. Very often, in a clay soil, the clay had been originally the same all through the surface soil, in the case of arable land being determined first by turf and later by the plough; but where a light loam rested on a chalk rock, there might be only 5 or 6 inches of actual soil, and if samples taken 2 or 3 inches deeper—and therefore including some of the chalk—were regarded as representing the top soil, there would obviously be a departure from what was rational and reasonable.

With regard to solvents, it was recommended by the committee that hydrochloric acid should be used for the determination of what was called, for the sake of brevity, "total" mineral constituents; and it was directed that the fine earth should be boiled with strong hydrochloric acid in an open flask, and digested on the ordinary water-bath for from forty to forty-eight hours, the flask being loosely stoppered. There was no suggestion that the solution should be evaporated to dryness. He (Dr. Dyer) thought that some reference to this point might well be introduced, because it was quite possible that some people might be found who, working under these directions, would assume that they were to use for the phosphoric acid determination an aliquot portion of that solution when filtered, without evaporation to dryness, to remove silica. The usual practice was to evaporate the whole thing to dryness—very often twice—in order to render all the silica insoluble, which, of course, was very essential for an accurate determination of phosphoric acid, and, indeed, of other constituents also. Unless the solution were sent to dryness, the silica would be more or less of a nuisance all through the analysis. He thought it should be pointed out that the aliquot portions of the solution might be afterwards evaporated to dryness, preliminarily to a further analysis, and the silica in that way removed. One difficulty in operating on the unignited soil was, that if a large quantity of organic matter was present, it was converted by the hydrochloric acid into more or less sugary substances, which, unless destroyed by evaporation to dryness with nitric acid, rendered very difficult the precipitation of the iron and alumina.

Of course, the great difficulty about ignition was, as the author had pointed out, that its effect on different soils was not the same. Very many clay soils (those at Rothamsted, for instance) contained nearly 2 per cent. of total potash, obtainable by

fusion, though only a small fraction of 1 per cent. was obtained by means of hydrochloric acid, even after ignition; and obviously the extent to which the silicates containing potash were decomposed depended a good deal upon (among other factors) the quantity of lime present in the soil. But even in the same soil the temperature and period of ignition affected to a considerable extent the quantities dissolved out afterwards. For getting uniform results it would appear to be better to work on the unignited soil, but precautions must be taken in some cases for the subsequent destruction of organic matter in the solution by boiling with bromine or by some other means. He had, after making a very great many careful and minute experiments, come to the conclusion that in the determination of phosphoric acid it did not matter at all whether hydrochloric or nitric acid was used as a solvent. The determination was virtually one of the total percentage present, and was therefore of a quite definite nature. If strong nitric or hydrochloric acid was used, with evaporation to dryness and redigestion, identical results were almost invariably obtained in either case; but the inconvenience of using hydrochloric acid lay in the fact that if the molybdic acid method was used, as it always would be, the presence of hydrochloric acid was a nuisance. It was almost necessary, for working with certainty and comfort, to evaporate to dryness with nitric acid, and so eliminate, or nearly eliminate, any chlorides present. A comparatively small quantity of hydrochloric acid in a determination by the molybdate method was apt to keep some of the phosphoric acid in solution, and while the precipitate was being washed, the filtrate would become turbid, and would have to be refiltered. This might to some extent be got over by the use of large quantities of ammonium nitrate; but there was really in his experience no reason for any hydrochloric acid being present, for a separate quantity of the solution could be taken for the phosphoric acid determination and evaporated to dryness, and redigested with nitric acid only.

He did not quite like the expression of the results on the air-dried soil. He quite believed in working on the air-dried soil, but he thought it would be more convenient to calculate the results on the soil dried at 100° C., because that was a definite basis. Air-dryness depended a great deal upon the condition of the atmosphere at the time of drying the soil, and upon the quantity of humus in the soil itself—a factor which varied considerably. The experience of Mr. Wood, that a larger quantity of potash was obtained after ignition, corroborated that of the late Dr. Voelcker, formed during an investigation made many years since on the effects of "paring and burning," which used to be a common agricultural operation, resulting in the liberation from clay soils of quantities of potash previously locked up from the reach of plants in the form of insoluble silicates. But in any case, when there was any clay in the soil, the potash extracted by either hydrochloric acid or nitric acid was only a small fraction of the total potash present, and it varied not only according to the way in which the soil was ignited, but according to the period of ignition and the strength of the acid used for extraction. In clay soils it was quite impossible, even using the same process, to get concordant results in the case of "total" potash determinations; but seeing that the quantity of potash extracted by strong mineral acids was always largely in excess of anything that was really available for plant use, it would not be very material whether one obtained 0·25 or 0·35 per cent., though it

was certainly desirable that, if this determination were made, the processes used should as nearly as possible be uniform.

He was naturally pleased to hear—he had been unaware of it until the previous day—that the process which he had described as an attempt to determine what might be called the "available" mineral matter in soils commended itself to the committee. An immense mass of work had been done on the subject, chiefly in America, since the publication of the paper in which the process was described. Solvents of all kinds had been tried—weak hydrochloric acid, weak nitric acid, weak oxalic acid, and various saline solutions. It was, however, probably not very material what weak acid solvent was used; the process must of necessity be a very arbitrary one, and, as far as he had been able to understand the American results, the citric acid process seemed to act as well as any other.

Dr. C. M. Luxmoore said that the chief object of the committee had been to deal with the question of soil analyses in connection with field experiments. The greater part of the work of soil analyses with which he had been connected at Reading was of a more thorough nature, and he thought that this would make some difference in regard to the question of depth. In the case of the soils (chiefly from the county of Dorset) which had recently been examined, a process had been adopted which probably would not always be practicable in the case of field experiments. Each sample was taken in three boxes, and displayed a section of the soil to a depth of 18 inches from the surface, so that the division could be made wherever seemed to be right. He had endeavoured, as far as possible, to insure that the surface soil should always be arbitrarily taken at some multiple of 3 inches. The advantage of this was that, if the surface soil actually analysed were taken down to, say, 6 inches, and then certain determinations were made in the soil from the lower depths, it was easy to calculate with considerable approximation the percentage composition of the first 9 inches of soil for purposes of comparison, even though the first 9 inches had not actually been analysed. If only one sample were taken to a certain depth, he thought it best to adhere, where possible, to the first 9 inches. Sir Henry Gilbert had expressed to him also the views referred to by Dr. Dyer, but he could not help thinking that the immense amount of work already done on 9-inch samples was a very strong argument against changing except for some definite reason. It was, however, provided in the report of the committee that any well-defined line of demarcation should be observed. With regard to the question of sieves, he would point out that the Rothamsted determinations made in the soil which passed through a ¼-inch sieve were not very much affected by that circumstance, because most of them had been determinations of nitrogen, and the nitrogen in the small stones or gravel introduced by this sieve was a small factor and more or less constant, the gravel being present in very similar proportion in the different plots. The questions of evaporation to dryness and of the use of the molybdate process were among the matters which had been deliberately left out of the report as being matters of analytical practice. It was the custom at Reading to take the hydrochloric acid solution and filter it right away; the insoluble residue was washed, dried, ignited, and weighed; and the filtrate evaporated to dryness and the residue redissolved. It seemed to be important that the solution should be filtered before being evaporated

to dryness. Evaporation to dryness before filtration has an irregular effect on the dissolution of the soil constituents, and this leads to a want of concordance in the results. In the course of evaporation of the filtered liquid to dryness it was easy to add a little nitric acid to destroy any organic matter. The phosphoric acid was always determined in a solution obtained from the precipitate given by ammonia, so that practically the chlorine was eliminated in that way. The ammonia precipitate, which included iron oxide, alumina, and phosphoric acid, was dissolved, an aliquot portion of the solution taken, and the phosphoric acid determined in it. The only serious objection which Dr. Dyer had taken to the recommendations of the committee was in regard to a point on which Mr. Hall and he (the speaker) felt very strongly, namely, the calculation of the results on the air-dried soil. At first sight it seemed as though drying in the water-oven was the more convenient basis to work upon, but he was disposed to doubt that. In one sense it was definite, but really the temperature of a water-oven, say 95° C., was perfectly arbitrary, as all soils lost further quantities of water on heating to a higher temperature. The air-dry condition seemed the more natural one to take as a basis, and therefore the only question was whether the air-dry soil was sufficiently definite. Of course, the soil gained and lost moisture day by day as it was exposed to the air, according to the temperature and the state of the atmosphere; but the variations were not at all considerable. Within the moderate limits of indoor temperature and of indoor atmospheric conditions as to moisture, they did not amount to more than about 1 per cent., which, calculated on the constituents of most importance, was comparable with the kind of error that could not possibly be avoided, namely, experimental error. He was not, of course, speaking of the difference between air-dried soil and soil dried at 100° C., but of the variations in air-dried soil itself depending upon the temperature indoors, which variations, as before stated, were not very great. In the case of the air-dried soil the analytical results would include the percentage of moisture, which was a rather useful item, as indicating the kind of soil that had to be dealt with.

Mr. EMBREY said that the author seemed not to have mentioned some important points in connection with what might be called the rough analysis of soils, such as the method of washing (which was by no means uniform), to estimate the fine sand and clay. He thought that in most parts of England it would be practically useless to adhere to a depth of 9 inches, the depth of the plough rarely exceeding 5 inches. In his own county (Gloucester) 7 inches was the greatest depth to which it was worth while going for any practical purpose. Any difficulty in this direction, however, might perhaps be got over by always stating the depth of soil taken. With regard to the calculation of results on the air-dried soil, in the case of some heavy soils that were very wet it might be a week before they were ready for analysis, while, on the other hand, a light soil might be ready in two or three days, and the question would arise as to how the time should be fixed when the calculation was to be made, a matter which would seem to be exceedingly difficult of determination.

Mr. HALL said that the point of most importance was perhaps that of depth. He could really only add to what had been said by Dr. Luxmoore, and had been indicated in the paper, that above all things the matter was one to be decided by each particular worker. When one was really seriously engaged upon the work of soil

analysis, taking one's own samples, it was sometimes necessary to take two or three samples from the same place at different depths if any trustworthy opinion was to be pronounced. But in cases where it was only possible to send instructions for taking a sample it was necessary to fix a depth. In his experience there existed a large number of soils (including some of the best soils in the country) showing no line of demarcation; and the committee wished rather to indicate that, if there was no guiding line to make one take any other depth, 9 inches should be taken. But, of course, any natural feature that might exist should not be disregarded. Certainly he agreed with Dr. Luxmoore that they did not wish to make any recommendations in regard to the determination of phosphoric acid and to the question of evaporation to dryness, which were essentially points for the analyst himself, who would recognise the necessity for getting rid of the silica and organic matter, but should be left to himself to decide as to the means of doing it. The committee had made a number of determinations to ascertain what were the variations in the weight of air-dried soil. A sample of soil was laid out on a table and weighed day after day under varying conditions; and, as Dr. Luxmoore had said, the widest variation was rarely as much as 1 per cent., which, on the final proportion of phosphoric acid, potash, or other constituent of importance, became negligable. He quite agreed with Dr. Luxmoore in regarding the air-dried soil as a more fixed entity than the soil dried at 100° C., or rather, at something between 95° and 100°, the temperature of the water-bath. If the temperature of drying were varied between 95° and 110°, it was possible to dry off varying quantities of moisture; and there was really no line of demarcation that could be drawn between water of combination and moisture that was merely mechanically retained, though he thought that the figure obtained for moisture in the air-dried soil was of some utility. Reference had been made to the question of mechanical analysis, but, speaking frankly, the committee had considered that point and had shirked it. There existed, so to speak, two diametrically opposed schools of procedure amongst mechanical analysts, the separation of the particles being effected in the one case by a current of moving water, and in the other case by decantation by the time factor; and he did not think, from his own reading, that the two were yet in line. Work was being done on this point by some of the members of the committee, but they were agreed that the time had not arrived for any joint recommendations.

ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

FOODS AND DRUGS ANALYSIS.

On the Instability of the Total Solids of Milk, and the Value of their Determination in Milk Analysis. A. Reinsch and H. Lührig. (*Zeit. für Untersuch. der Nahr. und Genussmittel*, 1900, iii., 521-531.)—The author refers to the fact pointed out by Vieth and others that from the time of milking the proportion of the total solids of milk undergoes a constant diminution. This diminution, he finds, is not accompanied by any decrease in the specific gravity of the milk, and he consequently infers that it must be due to the production and loss of volatile bodies during the processes of evaporation and drying, a view which finds confirmation in the fact that, on distilling milk which has been kept, volatile acids and other bodies are found in the distillate. The author shows, however, that if the total solids be determined by calculation from the specific gravity and the percentage of fat by Fleischmann's formula, then the result is accurate up to the time of curdling, since neither of these factors is liable to material alteration during this time.

Referring to the question of the determination of the specific gravity of milk serum as a means for the detection of added water, the author observes that in the summer months samples frequently reach the analyst in a curdled condition, rendering only an approximate determination of the fat possible, and that in such cases the specific gravity of the serum forms the only reliable indication. From a series of experiments made at different times of year, the author concludes that no sufficient change takes place in the serum within three days from the time of curdling to affect the accuracy of such deductions. H. H. B. S.

The Detection of Abrastol in Wine. A. Sanna Pintus. (*Staz. Speriment. Agrar. Ital.*, 1900, xxxiii., 274-277.)—Abrastol, or asaprol-etrasol, is the calcium salt of β-naphthol-β_3-sulphonic acid, and is used as a clarifier and antiseptic in wines. It is a white, inodorous neutral powder, with a bitter taste, and is soluble in water and alcohol, but not in ether.

To detect it, the author decolorizes a few c.c. of the wine with animal charcoal in the cold, and adds an equal volume of a solution of mercuric nitrate containing nitrous acid. An immediate yellow coloration, with a golden fluorescence, is obtained in the presence of abrastol; on exposure to the air for a few minutes the colour changes to rose. Since part of the abrastol is retained by the animal charcoal, an excess of the latter must be avoided. Usually 2 grammes will be found sufficient to decolorize 10 c.c. of wine. In the case of white wines, with which no treatment with charcoal is required, the test is capable of detecting 0·01 per cent. of the antiseptic.

The reagent is prepared by dissolving 100 grammes of mercury in 97 c.c. of nitric acid (42° Bé), and adding 35 c.c. of water to the solution. C. A. M.

ORGANIC ANALYSIS.

On the Separation of Oleic Acid from other Unsaturated Acids. K. Farnsteiner. (*Zeit. für Untersuch. der Nahr. und Genussmittel*, 1900, iii., 537-539.)—The author takes exception to Lewkowitsch's criticism of his method for the separation of oleic acid as barium oleate (ANALYST, 1900, xxv., 64), on the grounds that Lewkowitsch's experiments were not made with pure oleic acid, and that the details necessary to render them exact and scientific are wanting. Referring to Table II. in Lewkowitsch's paper, he remarks that it is not clear whether the figures denote the quantities of the barium salt dissolved in percentages of the whole, or whether they refer to the quantity of the barium salt contained in 100 parts of the benzene solution. H. H. B. S.

Determination of Carbonates and Alkali (Free and Total) in Soap. R. Henriques and O. Mayer. (*Zeits. angew. Chem.*, 1900, 785.)—The author determines the carbon dioxide gravimetrically, using an excess of $\frac{N}{2}$ hydrochloric acid. The residue is then raised to the boil, the fatty acids filtered off, and washed till neutral. The aqueous liquid is titrated with seminormal soda to give the total alkali; the fatty acids are dissolved in warm alcohol and titrated with alcoholic seminormal alkali to give the combined alkali. If, however, the soap has been made with oils containing fatty acids soluble in hot water, the foregoing process becomes more or less inexact. In this case the total acids and the true soap must be determined separately by decomposing the sample in a stoppered funnel with hydrochloric acid, extracting the fatty acids with ether, washing them, titrating with alcoholic alkali, and weighing the soap so reproduced. F. H. L.

The Composition of Jasmine Oil. Jeancard and Satie. (*Bull. Soc. Chim.*, 1900, xxiii., 556, 557.)—Referring to the investigations of Verley and of Hesse and Müller (ANALYST, xxv., 46), the authors point out that most chemists studying this oil have extracted it from the pomade, and that their results are therefore liable to error, for the lard or beef-fat forming the basis of the pomade is prepared by being heated with jasmine water, and sometimes with rose water and neroli water, and is preserved by an addition of benzoin or alum.

Thus, the authors have found in a kilo of jasmine pomade 0·05 gramme of benzoin, 0·25 gramme of neroli oil, and 3·0 grammes of jasmine oil. On extracting the pomade with the usual solvents, foreign substances are dissolved to a considerable extent, and may compose about 11 per cent. of the oil finally obtained.

In order to obviate this, they have extracted the oil by *enfleurage* in the usual manner, but substituting untreated vaseline for the prepared fat.

Two samples thus obtained gave the following results:

	Specific Gravity at 17·5° C.	Rotatory Power $l=20$ millimetres.	Saponification Value.	Esters as Linalyl Acetate.	Esters as Benzyl Acetate
I.	0·9099	+0° 30′	103·6	36·26	27·70
II.	0·9201	+0° 32′	120·0	44·10	33·75

Two oils obtained by distillation of the flowers in a current of steam were also examined, Sample III. being prepared directly from the flowers, and IV. distilled from the residue left from the *enfleurage*.

	Specific Gravity at 17·5° C.	Rotation $l = 100$ millimetres.	Saponification Value.	Esters as Linalyl Acetate.	Esters as Benzyl Acetate.
III.	0·9246	+1° 40′	155	54·39	41·65
IV.	0·8900	+3° 50′	77	26·95	20·62

C. A. M.

The Examination of Oil of Lemon. G. Fabris. (*Annali del Laboratorio Chim. Cent. delle Gabelle*, 1900, iv., 53-121.)—According to the author, the substances most generally employed to adulterate oil of lemon are turpentine, with oil of orange, to raise the rotatory power of the mixture to that of the normal oil. He gives the following table of the rotation of different samples in a 100-millimetre tube at 20° C.:

	$[\alpha]_D$
Oil of turpentine, French	−20°
,, ,, common	−5° 30′
,, ,, American	+4° 30′
Oil of lemon, distilled	+52° 15′
Lemon terpenes, distilled in laboratory from pure oil ...	+60° 40′
Terpenes of Messina lemons	+55° 50′
Limonene (Schimmel and Co.)	+102° 4′
Oil of orange	+97° 10′
Oil of lemon, pure	+60° 42′

From a long series of experiments with mixtures of these substances the author has arrived at the conclusion that there is no method in existence capable of detecting every extraneous addition to oil of lemon; although in his opinion the best means of detecting the most common and probable form of adulteration is the method of fractional distillation as described by Schimmel and Co. (*Report*, October to November, 1896), and that of Soldaini and Bertè (ANALYST, xxv., 104). He considers that whilst these methods only give vague indications as to the nature and amount of foreign substances added to the oil, and cannot detect oil of turpentine in small proportion, or lemon terpenes if not added in large excess, yet that they are capable of detecting 5 per cent. of oil of turpentine (or less of lævorotatory turpentine), and the presence or absence of oil of orange can be inferred from the rotation of the distillation residue.

Other analytical tests, such as the determination of different physical and chemical constants, and various colour reactions, are, in his experience, less sensitive, though they should not be omitted. Of the colour-tests the following are specially recommended:

The Guaiacum Test.—In this test (Hager, *Seif. Oel. u. Fette*, 1898, 179) a pinch of powdered guaiacum resin is placed in a test-tube, into which are then introduced 5 or 6 c.c. of alcohol and 0·5 c.c. of the oil of lemon; or better, 0·2 gramme of

guaiacum resin is dissolved in 100 c.c. of alcohol (about 95 per cent.), and 6 or 8 c.c. of a solution containing 0·5 c.c. of the oil added. On boiling the contents of the tube for a moment, the liquid assumes a brown tint, which soon changes to violet in the case of pure lemon oil. With oil of turpentine a pronounced violet is observed. When left for fifteen minutes and then diluted with 10 c.c. of alcohol, the liquid containing oil of lemon becomes faint brown; with turpentine it assumes an intense blue colour; and with a mixture a faint but distinct blue tint. A comparative test should be made with an oil of lemon of known purity.

The author states that old or badly kept lemon oil tends to give a blue colour, and that French turpentine oil reacts much more rapidly than American turpentine.

Oil of orange does not give any coloration with guaiacum resin.

Potassium Iodide Test (Marpmann).—When turpentine is dropped on to a crystal of potassium iodide, a black coloration is obtained after some time, whilst lemon oil gives a yellow colour, and mixtures a brown shade. According to the author, a mixture containing less than 5 per cent. of turpentine does not differ materially from pure oil of lemon as regards the colour produced.

Amyl Nitrite Test.—Two c.c. of the oil are treated with 6 to 8 c.c. of 96 per cent. alcohol, and after the introduction of 10 or 12 drops of ethyl or amyl nitrite, and 2 or 3 drops of hydrochloric acid (specific gravity, 1·17) the liquid is boiled for an instant and then left to cool. With lemon oil a yellow or orange colour is obtained; with turpentine a rose to blood-red colour; and with mixtures a rose coloration varying in shade with the proportion of turpentine. The author found that a mixture containing 2 per cent. of French turpentine oil gave a marked rose colour, but that in the case of American turpentine the test was not sensitive for less than 5 per cent.

C. A. M.

The Composition and Analysis of Oil of Bergamot. G. Fabris. (*Annali del Laboratorio Chim. Cent. delle Gabelle*, 1900, iv., 123-136.)—This oil is expressed almost exclusively in the province of Reggio Calabria, near the coast, the machinery used being of the most primitive type. The yield of oil varies with the season, locality, and maturity of the fruit, but in the mean 1,500 bergamots are required to produce 1 kilo. of the oil. The best and most valuable oil is that obtained from the ripe fruit in January. The oil which separates from the pressed liquid has a more or less pronounced yellowish-green colour, whilst that recovered from the aqueous part of the pressings is light yellow, and has a special odour due to the heat.

The oil expressed from unripe or damaged fruit which has fallen from the trees is known as *Nero of Bergamot.* It has a brownish-green colour and an odour which is not very fragrant.

The principal constituents of bergamot oil are terpenes (among which have been identified limonene and bergaptene) and linalyl acetate. The specific gravity of the pure oil varies from 0·882 to 0·886 at 15° C., according to Schimmel and Co. (*Report*, 1896, ANALYST, xxiii., 13), but according to Campolo (*Staz. Sper. Agrar. Ital.*, 1895, xxviii., 433) it should be 0·887. The rotatory power in a 100 millimetre tube at 15° to 20° C. varies from +8° to 20°. When distilled by Soldaini and Bertè's method (ANALYST, xxiv., 50; xxv., 104), it boils at 74° C. to 120° C. under a pressure

of 20 to 30 millimetres, and the rotatory power of the successive fractions diminishes from right to left. The residue (about 7 per cent.) is a semi-solid mass.

The amount of linalyl acetate is variable. Schimmel and Co. give 34 to 48 per cent., with a mean of 33 per cent.; Helbing and Passmore 42·4 per cent.; and Campolo (*loc. cit.*) 37·57 to 39·87. It is usually accepted that a good oil should contain about 39 per cent. (*cf.* Bornträger, ANALYST, xxi., 137; and xxii., 41).

The amount of esters varies with the kind of season, locality, and maturity of the fruit. Thus, the oil from green bergamots was found to contain 31 per cent., that from half-ripe fruit 33·2 per cent., and that from over-ripe fruit 37·7 per cent. (Schimmel and Co.'s Report, April, 1896; *cf.* Charabot, *Comptes Rend.* cxxix., 728).

Nero of bergamot and distilled oil are much poorer in esters than good expressed oil, the first containing only 23 to 25 per cent., and the second 12 per cent. as the maximum.

Seven specimens of good oil examined by the author gave the following results: Specific gravity at 15° C., 0·8860—0·8870; rotation at 20° C., +11° 35′ to +15°; linalyl acetate, 34·10 to 41·00 per cent.; residue on evaporation 4·0 to 5·55 per cent., containing 1·05 to 2·05 per cent. of linalyl acetate. All were soluble in 90 per cent. alcohol.

A sample of distilled oil showed on analysis: Specific gravity at 15° C., 0·8700; rotation at 20° C., 30° 15′; linalyl acetate, 10·15 per cent.; residue on evaporation 1·45 per cent.; linalyl acetate in residue 0·50 per cent.

With a sample of Nero of bergamot the corresponding figures were: Specific gravity, 0·885; rotation at 20° C., +9° 30′; linalyl acetate, 25·50 per cent.; residue on evaporation 2·70 per cent.; and linalyl acetate in residue 1·75 per cent.

The substances used to adulterate bergamot oil are very numerous, and include fatty oils, resins, Canada and Gurjun balsams, turpentine, oils of lemon, orange, and cedar-wood, and the distillation products of bergamot oil itself.

Of these, fatty oils, resins, and balsams increase the specific gravity and residue on evaporation. Resins, especially colophony, increase the proportion of esters and the acidity of the oil, normal bergamot oil being only very slightly acid. The residue of a pure oil on evaporation should not exceed 6 per cent. Fixed oils and resins will be indicated by the saponification value of the residue, and the former, with the exception of castor-oil, by the solubility-in-alcohol test.

The author shows by the figures obtained with experimental mixtures that 15 per cent. of turpentine can be added to bergamot oil without causing the constants to deviate to any extent from their normal limits. Soldaini and Bertè's method of fractional distillation affords a valuable criterion, but even with it a notable quantity of dextro-rotatory turpentine may escape detection.

Oil of lemon and oil of orange increase the rotatory power and diminish the specific gravity, quantity of esters, and residue left on evaporation. Indications of such an addition will be furnished by Soldaini and Bertè's method of fractional distillation. Pure bergamot oil, unlike orange and lemon oil, gives no coloration with Schiff's reagent (absence of aldehydes). Moreover, on testing 2 or 3 c.c. of pure oil of bergamot in 10 to 15 c.c. of absolute alcohol, with a small quantity of metaphenylenediamine hydrochloride, the liquid does not change colour, or only

becomes slightly yellow; if, however, oil of lemon be present, a deep yellow or brown colour is produced. A comparative test should be made with bergamot oil of known purity.

An addition of the distillation products of bergamot oil lower the specific gravity, the percentage of esters, and the residue on evaporation. In the author's opinion, however, they can be added in considerable quantity without materially changing the characteristic properties of a pure oil. C. A. M.

The Causes of the Variation in the Amount of Esters in Oil of Lavender. Jeancard and Satie. (*Bull. Soc. Chim.*, 1900, xxiii., 549-554.)—As the commercial value of lavender oil depends upon the amount of esters it contains, the authors have examined a large number of samples in order to determine what influence the altitude, the maturity of the plant, and the process of distillation have on the composition of the oil.

Eleven samples of oil were prepared from flowers gathered in the same district, at the same time, and under the same conditions, but at different altitudes, varying from 1,000 to 1,800 metres. From the results the authors conclude that the altitude has no influence on the physical properties of the oil.

The oil extracted from the flowers only was found to be far superior to that obtained from the leaves and stems. The comparative analytical results were:

	Yield per 1,000 Kilos.	Specific Gravity at 26° C.	Rotation at 26° C., 100 m.m.	Esters, per cent.	Soluble in 70 per cent. Alcohol.
Oil from leaves and stems	0·500 k.	0·8850	− 7° 12′	41·65	2·4
Oil from flowers	1·985	0·8814	− 6° 40′	39·44	2·4

As regards the influence of the method of extraction, the authors show that if the distillation be too rapid the whole of the esters are not obtained. Moreover, during the distillation there is a partial saponification of the esters, which are reduced from, say, 39 to 35·5 per cent. In order to determine the conditions of this saponification, lavender oil was boiled with twenty times its weight of distilled water (this being about the proportion used in actual practice) under a reflux condenser, and the percentage of esters determined from hour to hour. It was found that the loss of esters from saponification amounts at first to about 2 per cent. per hour, but gradually diminishes towards a point of equilibrium where the saponification is compensated by an equal acetylation.

The amount of saponification is proportionately increased by the presence of carbonates in the water. C. A. M.

The Estimation of Carvol. H. Labbé. (*Bull. Soc. Chim.*, 1900, xxiii., 280-286.)—For the determination of carvol in the presence of limonene and other hydrocarbons which normally accompany it, the author describes two methods. These are based upon the facts that carvol readily forms a dihydrogen disulphonate which is soluble in a solution of bisulphite, whilst limonene is not soluble to any appreciable extent in a boiling solution of sodium bisulphite, nor does its presence interfere with the carvol reaction.

I. Five grammes of the mixture are boiled under a reflux condenser with about 15 grammes of sodium bisulphite and some sodium carbonate. After about an hour and a half the contents of the flask are cooled, and the supernatant oil extracted with anhydrous ether, the neck of the flask and the condenser being washed with the same solvent. The ethereal extract is separated, dried over anhydrous sodium sulphate, and filtered into a small weighed flask fitted with a tube 15 centimetres in length to act as a condenser. After the greater part of the ether has been evaporated under reduced pressure, the last traces are removed by warming the flask. The weight of the residue subtracted from the weight of the mixture taken gives the quantity of the carvol.

The following experiment may be quoted in illustration of the accuracy of the method: Taken limonene, 4 grammes (80 per cent.); carvol, 1 gramme (20 per cent.). Found limonene, 3·976 grammes (79·52 per cent.).

In one experiment, in which the mixture was boiled for five hours, the amount of limonene was 2 per cent. too low. Under the conditions mentioned above, the error, which is invariably less than 1 per cent., is attributed to an inevitable loss of limonene in the manipulation.

II. From 1 to 1·5 gramme of carvol were boiled with 10 c.c. of a standardized solution of sodium bisulphite containing 0·2 to 0·3 gramme per c.c. The sulphur dioxide liberated in the reaction was collected in a slightly alkaline solution, which was subsequently titrated with a standard solution of iodine. The residue in the flask was also titrated, and the total amount of sulphur dioxide found was deducted from that in the sodium sulphite originally taken. The amount of sulphur dioxide taken up by the carvol was calculated into carvol by using the formula $C_{10}H_{14}O(SO_3NaH)_2$ for the dihydrogen disulphonate.

In this test experiment the quantity of carvol found was 100·3 per cent. In another commercial sample the carvol amounted to 96·9 per cent. C. A. M.

Estimation of Metacresol in Presence of its Isomers. F. Raschig. (*Zeits. angew. Chem.*, 1900, 759.)—"Cresylite," a product made by nitrating cresol, has been used for years as a substitute for "lyddite," etc., in the manufacture of explosive shells. This substance consists of trinitro-*m*-cresol; for when a mixture of the three cresols is treated with hot nitric acid, only the meta-compound is nitrated, the others being oxidized to oxalic acid. In the preparation of cresylite, therefore, *o*- and *p*-cresol cause a waste of acid, and accordingly, to the manufacturer, the mixture is the more valuable the higher the percentage of *m*-cresol it contains. The author's process is based on the above reaction with nitric acid. Exactly 10 grammes of the sample, which should distil between 190° and 200° C., are weighed out into a small flask, mixed with 15 c.c. of sulphuric acid of 66° Beaumé, and placed for an hour in a steam-heated oven. The product is then poured into a wide-necked flask which holds at least 1 litre, and is made to form a thin coating on the walls of the vessel by rotating it under a cold-water tap. Into the sulphonating flask are next brought 90 c.c. of nitric acid, 40° Beaumé, and by agitation the residue of sulphonic acids is dissolved. The solution is then poured quickly into the large flask, and the whole is

shaken thoroughly for about twenty seconds till the sulphonated cresols have dissolved. The vessel is placed in a draught cupboard until the reaction is finished; the liquid becomes turbid and clear again, when oily drops of trinitrocresol separate. After waiting five minutes, the mass is poured into 40 c.c. of water contained in a basin, and the flask rinsed with the same volume of water. The whole is set aside for at least two hours till quite cold, the solid matter broken up with a rod, and filtered with the pump. The precipitate is washed with 100 c.c. of water, dried on the paper at 95° to 100° C., and weighed. It is most important not to decrease the quantity of nitric acid prescribed, to add it rapidly and all at once to the sulphonic acids; to use a large flask for the nitration, and to see that the neck is sufficiently wide.

The process is accurate within 1 per cent. in the presence of either or both *o*- and *p*-cresol, yielding 174 per cent. of trinitro-*m*-cresol. The presence of 10 per cent. of phenol does not affect the results, for the picric acid remains in solution. A larger proportion of phenol is not likely to be met with commercially, but it would be shown by a lower boiling-point of the original sample, or by the nitro-derivative softening or melting when heated to 95° C. Xylenols would also cause the nitro-product to melt on warming or to remain liquid on cooling. F. H. L.

The Tragacanth Method of assaying Alkaloids. J. B. Nagelvoort. (*Amer. Journ. Pharm.*, 1900, lxxii., 270-272.)—This method is slightly modified from the original one of Rusting, and is recommended for the rapid and accurate assay of fluid extracts. Three grammes of the extract are mixed with 5 c.c. of water in a small dish and the alcohol evaporated. The residue is transferred to a 100 c.c. flask, 60 c.c. of ether added, and the solution rendered alkaline. A 5 or 10 per cent. solution of sodium hydroxide is next introduced in successive small quantities, care being taken to avoid having an excess of alkali. The flask is then well shaken for about a minute, 2 grammes of powdered tragacanth introduced, and the contents again shaken. After standing for some time, 40 c.c. of the clear supernatant liquid are transferred to an Erlenmeyer flask of known weight, the ether distilled, and the residue dried at 50° C. with the aid of a current of air supplied by a rubber "spray" ball.

For the assay of quinine tannate 1·5 grammes are mixed with a little water, 75 c.c. of ether introduced, the liquid made alkaline with sodium hydroxide, shaken with tragacanth as above, and 50 c.c. of the supernatant liquid evaporated.

Fluid Extract of Hydrastis.—This is assayed by diluting 10 grammes with 20 c.c. of water in an Erlenmeyer flask, and slowly evaporating down to less than 20 grammes. The liquid is then cooled, made up to 20 grammes in weight, shaken with some kieselguhr, and filtered. Ten grammes of the filtrate are transferred to a flask, 25 c.c. of ether added, and afterwards 3 c.c. of 10 per cent. ammonium hydroxide, and the whole well shaken. The shaking is repeated after the addition of 25 c.c. of petroleum spirit, and, lastly, 2 grammes of powdered tragacanth are introduced and the contents of the flask shaken once more and left to settle.

Forty c.c. (= 4 grammes of fluid extract) of the clear liquid are removed, and the

ether evaporated by rotating the flask in water at 30° to 35° C. for a few minutes. The flask is then cooled, well corked, and placed in a freezing mixture for the hydrastine to crystallize out. The supernatant liquid is decanted, and the crystals dried and weighed in the usual manner. C. A. M.

The Detection of Urobilin in Urine. T. Roman and G. Delluc. (*Journ. Pharm. Chim.*, 1900, xii., 49, 50.)—The ordinary test with an alcoholic solution of a zinc salt may be simplified in the following manner: 100 c.c. of the urine are acidified with 8 to 10 drops of hydrochloric acid and shaken with 20 c.c. of chloroform. After separation of the liquids, about 2 c.c. of the chloroform layer are tested with 4 c.c. of a solution of 1 gramme of crystallized zinc acetate in a litre of 95 per cent. alcohol. At the junction of the two layers the green fluorescent ring characteristic of urobilin will appear, and on shaking, the fluorescence, which is rose-coloured by refracted light, will be distributed throughout the liquid.

It was found that certain commercial alcohols produced the fluorescence without any addition of a zinc salt, but this was proved to be due to the presence of traces of zinc probably derived from the vessels in which the alcohols were rectified.

C. A. M.

Determination of the Softening Point of Pitch. J. Klimont. (*Zeits. angew. Chem.*, 1900, 761.)—Schenk has described a process according to which powdered pitch is put into a tube and a drop of mercury brought on to the surface. The whole is warmed, and the temperature at which the pitch swells is called the melting-point, while the temperature at which the mercury sinks into the pitch is called the liquefying-point. The test requires some experience to carry out successfully, for the powder coats the drop of mercury, and prevents the operator seeing it. The two following methods are better: Finely-powdered pitch is run into a test-tube through a long funnel until, after consolidation by tapping on the bench, the hemispherical bottom is just full. The tube is fastened to a thermometer and brought into a water-bath (a glycerin-bath if the sample is "hard"). The liquid is warmed slowly with a 2 centimetre flame. The softening-point is taken when the dust on the walls of the tube agglomerates into minute drops, the liquefying-point when the whole surface of the pitch becomes smooth and brilliant. In the other process some of the sample is softened and kneaded into a little rod 5 millimetres long and 3 millimetres in diameter. This is held with tongs, one point melted, inserted into a test-tube, and made to adhere to the bottom, so that it stands up vertically. The tube is filled with mercury till the rod is just covered, placed in a water- or glycerin-bath and warmed gently. Directly the rod rises and shows a black spot on the mercury the softening-point is read off; when the point broadens and becomes a drop the liquefying-point is noted. All three processes yield practically identical figures.

F. H. L.

INORGANIC ANALYSIS.

Electrolytic Determination of Lead. C. Marie. (*Bull. Soc. Chim.*, 1900, xxiii., 563, 564.)—The principal methods of separating lead are based on its precipitation as sulphate or sulphide, and the latter is converted into sulphate by treatment with nitric acid.

Lead sulphate is soluble in nitric acid containing ammonium nitrate, and the solution for electrolysis is prepared in the following manner: The sulphate is placed in the vessel of Bohemian glass in which the electrolysis is to be carried out, and heated on the water-bath with nitric acid, to which a few crystals of ammonium nitrate are added. When the sulphate has been completely dissolved, the solution is diluted with hot water and electrolysed in the usual manner at a temperature of 60° to 70° C. About 5 grammes of ammonium nitrate are required to dissolve 0·3 gramme of lead sulphate, and the nitric acid should be used in such proportion that the liquid after dilution contains 10 per cent. of free acid. With an electrode of roughened platinum, 90 sq. c.m. area, and a current of 0·3 A, 0·4 gramme of lead dioxide should be deposited in three hours.

The method can also be used in the analysis of glass containing lead; the finely powdered glass being treated with hydrofluoric acid and sufficient sulphuric acid to convert the bases into sulphates, and the lead sulphate dissolved as described above.

C. A. M.

The Determination of Arsenic in Metals and Alloys. A. Hollard and L. Bertiaux. (*Bull. Soc. Chim.*, 1900, xxiii., 300-302.)—The advantages claimed by the author for the following method are that it can be employed in the presence of antimony, that it is very rapid, and that only one distillation is necessary.

The arsenic chloride which distils over on boiling 5 grammes of the sample with 50 grammes of ferrous sulphate and 150 c.c. of hydrochloric acid is conducted through a U-tube containing some glass balls, heated to a temperature of 150° to 175° C. in an oil-bath, and collected in a cylinder containing 50 c.c. of water. Any antimony present remains in the distillation flask or the U-tube. The distillation is stopped as soon as 35 c.c. of liquid have passed over, and the cooled distillate titrated with a standard solution of iodine.

C. A. M.

The Analysis of Chrome and Tungsten Steels. A. G. M'Kenna. (*Proc. Engin. Soc. of West. Pennsylvania*, 1900, 119.)

Sulphur.—Five grammes are rapidly dissolved in hydrochloric acid, and the evolved gases are passed into an ammoniacal cadmium chloride solution, the last traces of gas being expelled by boiling. The sulphur is then determined by titration with iodine.

Silica and Tungsten.—The iron solution from the above is evaporated to dryness first with nitric acid and again with hydrochloric. The solid mass is then dissolved in hydrochloric acid, and the silica and tungstic acid filtered off, washed, ignited in a platinum crucible, and weighed. A few drops of hydrofluoric acid are now added, and the crucible is heated to bright redness for five minutes to volatilize silica. The

residue is WO_3, but may contain a little iron, which must be determined by fusing with sodium carbonate, dissolving in water, and filtering off the iron precipitate and weighing in the usual way.

Manganese.—The filtrate from the above is evaporated to small bulk, 50 c.c. of concentrated nitric acid are added, and the solution is boiled till all the hydrochloric acid has been removed. It is then made up to 200 c.c. with concentrated nitric acid and heated to boiling, 10 grammes of potassium chlorate are added, and the solution is boiled down to 75 c.c. The manganese is thus entirely precipitated as MnO_2. This is filtered on an asbestos plug whilst hot, and washed a few times with freshly-boiled concentrated nitric acid. It is redissolved in hydrochloric acid with a little potassium nitrite, the solution is freed from iron by means of ammonia, and the manganese is precipitated with bromine and weighed as Mn_3O_4.

Chromium.—The filtrate from the manganese contains the chromium as chromic acid. The filtrate is cooled and diluted to about 500 c.c. Excess of standard ferrous sulphate solution is then added and titrated back with permanganate.

Phosphorus.—A weighed portion of steel is dissolved in nitric acid with the addition of hydrochloric acid. The solution is evaporated to dryness, ignited, taken up again with hydrochloric acid. The silicon and tungsten are filtered off, 35 c.c. of strong ammonia are added, then a slight excess of nitric acid, finally 100 c.c. of molybdate solution. The flask is well shaken, allowed to stand an hour. The precipitate is then filtered on to a tared filter, washed with 1 per cent. nitric acid, dried for an hour and weighed. The phospho-molybdate contains 1·63 per cent. phosphorus.

Carbon.—The carbon is separated by means of chloride of copper and potassium. It is burnt in oxygen, the carbon dioxide is absorbed in barium hydrate solution, and the barium carbonate is filtered off and weighed. A. M.

A Rapid Method for the Determination of Carbon in Iron or Steel by Combustion. G. W. Sargent. (*Journ. Amer. Chem. Soc.*, 1900, xxii., 277.)—The combustions are performed in a porcelain tube heated in a three-burner furnace. This is not allowed to cool between two determinations; as soon as one is completed, the platinum boat is removed and another containing a fresh carbon residue inserted in its place. The products of combustion are passed first through moist sand, to retain hydrochloric acid or chlorine, next through a red-hot copper tube containing copper oxide, then through a spiral immersed in water to cool the gas, and finally through 6 inches of dehydrated calcium chloride. The carbon dioxide is then absorbed in potash bulbs as usual. The air and oxygen used are first purified by passing through a red-hot copper or platinum spiral, next through potash bulbs, then through an empty tube, and finally into the porcelain tube. The ends of this spiral, as also of the copper tube, are kept cool by means of a current of water circulating round them. The combustion is performed for the first ten minutes with oxygen at the rate of 4½ bubbles per second, then for further ten minutes with air at 5 bubbles per second. The combustion is then complete, and a fresh one is at once started. Twenty-five combustions per day can thus be done in one apparatus. A. M.

On the Analysis of Chrome-Iron Ore by the Borax Method. R. W. Emerson MacIvor. (*Chem. News*, lxxxii., 97.)—This note is occasioned by the publication of R. Fieber's paper on "A Simple Method of decomposing Chromite" (*Chem. Zeit.*, xxxiv., 333, and *Amer. Journ. Sci.*, 1900, x., 55), in which the following method is described: 0·5 gramme of the ore is fused in a platinum crucible with 3 grammes of sodium potassium carbonate for ten minutes; when cold, 3 grammes of borax are added, and the whole again fused, at first over a Teclu burner, then for forty-five minutes over the blow-pipe. If decomposition is incomplete, more of the mixed carbonate is added, and the fusion repeated. This process does not materially differ from that given in fairly old text-books, and leaves uncertain how many fusions are necessary to decompose the ore. A process used by Professor Dittmar and the author, and further improved by Dittmar alone, does not suffer from this defect, the undecomposed ore rarely exceeding 2 milligrammes per gramme of ore. These small quantities may be assumed to contain 50 to 60 per cent. of Cr_2O_3 without serious error. The outline of the process is as follows: 4 grammes of a flux, composed of equal weights of borax glass and $NaKCO_3$, previously melted together, are fused in a platinum crucible over an Iserlohn lamp, allowed to solidify, and 0·5 gramme of ore added. The covered crucible is then heated till the ore sinks to the bottom, then inclined, the lid being placed horizontally in front, and heated, whilst the contents are stirred with a platinum wire. After half an hour's heating, the melt is allowed to solidify, 2·5 grammes of $NaKCO_3$ added, and the whole again fused. The cold melt is extracted with hot water, alkaline chromate passing into solution. For a complete analysis it is best to work on 1 gramme of ore. A. G. L.

The Determination of Iron in Puddle Slags. L. Blum. (*Zeit. anal. Chem.*, 1900, xxxix., 156, 157.)—The ordinary volumetric methods used for this determination invariably give too high results in the presence of vanadic acid, (a frequent constituent of slags), owing to the fact that vanadic acid is reduced by stannous chloride or nascent hydrogen, and that the vanadium salts are oxidized by potassium permanganate or potassium bichromate.

In order to avoid this source of error and to obtain absolutely reliable results, the author recommends the following method of analysis: The solution of 0·5 gramme of the substance, containing the iron in the form of chloride, is freed from silica, neutralized with ammonium carbonate, and boiled with ammonium acetate. The precipitate is filtered off, dissolved in hydrochloric acid, and reprecipitated, in order to effect a complete separation from manganese. It is again dissolved, tartaric acid added, and made alkaline with ammonium hydroxide. An excess of ammonium sulphide is then added, and the beaker left for some time in a warm place. The vanadium sulphide and any small quantities of phosphoric acid, aluminium or calcium remain in solution.

The precipitated iron sulphide is filtered off, washed with water containing ammonium sulphide, and dissolved in dilute hydrochloric acid. The solution is oxidized with nitric acid, any precipitated sulphur separated, and the iron precipitated with ammonium hydroxide, ignited, and weighed as ferric oxide.

Although this method is tedious, the author regards it as the only one applicable to the determination of iron in the presence of vanadium and manganese.

C. A. M.

The Estimation of Manganese as Pyrophosphate. W. Böttger. (*Berichte*, 1900, xxxiii., 1019-1022.)—In Autenrieth's modification of Gibbs' method of determining manganese there is a risk of the precipitated salt being oxidized by the atmospheric oxygen, and to obviate this the author recommends the following modification: A quantity of an ammonium salt is added in the proportion of five to ten molecules to each molecule of manganese salt, which must be present in a neutral solution, this is then heated to the boiling-point, and treated with an excess of a solution of di-sodium phosphate. The hydrochloric acid which results from the reaction is neutralized with ammonium hydroxide, and the liquid boiled until the amorphous precipitate of manganese ammonium phosphate becomes crystalline. The precipitate is washed with cold, slightly ammoniacal water, and ignited in a blow-pipe flame or Hempel's oven until free from ammonia.

The results obtained in this way by the author varied from 99·8 to 100·4 per cent. of the theoretical. C. A. M.

The Separation of Nickel and Cobalt. O. F. Tower. (*Journ. Amer. Chem. Soc.*, 1900, xxii., 501.)—The author has made an investigation of the causes of the failure of Villiers' process for the qualitative separation of nickel and cobalt (*Compt. rend.*, cxix., 1263, and cxx., 46). The method as commonly carried out is as follows: The solution of the sulphides of the two metals in *aqua regia* is evaporated to dryness, and after suitable dilution sufficient tartaric acid is added to prevent precipitation by sodium hydroxide. This last reagent is then added until the solution is strongly alkaline, and hydrogen sulphide is added to saturation. Cobalt sulphide is precipitated, while nickel sulphide remains in solution, imparting to the solution a dark colour. When nickel is present only in very small quantities the colour is brown; with larger quantities it is a jet black.

The oxidation of the solution results, however, in the separation of so much sulphur that if nickel is present and cobalt is not, the black solution colours the sulphur, making it very difficult to distinguish it from precipitated cobalt sulphide. Furthermore, according to the process used in this separation, sodium chloride is present in the solution, and although this aids in the complete precipitation of cobalt sulphide, it may cause some nickel sulphide to be precipitated which in the absence of cobalt sulphide may be mistaken for it. A. M.

Determination of Cobalt in Presence of a Large Excess of Nickel. T. Moore. (*Chem. News*, 1900, lxxxii., 73.)—The solution containing the chlorides of the two metals and free from excess of acid is treated, if necessary, with zinc oxide to remove iron, and filtered. The filtrate is diluted to 300 or 400 c.c., 1 drop of hydrochloric acid added, and brought nearly to the boiling point. Bromine water and a moderate excess of zinc oxide are next introduced, the whole boiled for five minutes to expel

the bulk of the bromine, filtered, and the precipitate washed thoroughly. It is then rinsed off the paper into a beaker; the paper is extracted with very weak hydrochloric and sulphurous acids, the solution evaporated to dryness, taken up in a few drops of water, and added to the main precipitate. The latter is well boiled to disintegrate it, allowed to cool, mixed with 5 or 10 c.c. of hydrogen peroxide rendered strongly alkaline with sodium hydroxide, boiled for two or three minutes, and cooled again. It is then digested in a closed bottle with potassium iodide and hydrochloric acid, and the free iodine titrated as already described (*cf.* Abst. in this vol., p. 279).

F. H. L.

On the Separation of Cobalt from Nickel by means of Persulphates. F. Macrow. (*Zeit. Anorg. Chem.*, xxv., 197.)—After the author had completed his work on the action of potassium persulphate on cobalt salts (*Zeit. Anorg. Chem.*, xxiv., 262), the patent of Salomon and Coehn on the separation of cobalt from nickel was published in the *Zeitschrift für Electrochemie* (vi., 43, 532). They state that all persulphates precipitate cobalt quantitatively from solutions of cobalt and nickel salts, and oxidize the cobalt only in a mixture of their hydroxides. As the author had found that potassium persulphate does not completely precipitate cobalt from its salts, and only oxidizes a part of the hydroxide, the other part going into solution, he now repeated his investigations, using a larger excess of persulphate and warming for a longer time, and also studied the action of the persulphate on nickel salts and hydroxide. Potassium persulphate was again the only persulphate used. The same results as before were obtained, no quantitative separation being possible. The precipitation of cobalt from solutions of its salts is not complete, whilst a very small quantity of nickel is also precipitated. If the hydroxides are used, only a part of the $Co(OH)_2$ is oxidized, the other part being dissolved. The same is the case with $Ni(OH)_2$, although this dissolves more easily than the former; on the addition of dilute acids to the oxidized precipitates, cobalt, as well as nickel, is found in solution.

No quantitative determinations were made, the qualitative tests being conclusive. An analysis of the blackish precipitate obtained from $Ni(OH)_2$, after drying over H_2SO_4 and $CaCl_2$, gave results indicating the composition $Ni_3O_4 \cdot 6H_2O$; but the substance retains persulphate, and is not perfectly homogeneous. A. G. L.

Preparation of Potassium Xanthate for Nickel Determinations. E. D. Campbell. (*Journ. Amer. Chem. Soc.*, 1900, xxii., 307.)—In 1895 the author with W. H. Andrews published a method for the determination of nickel in nickel steel (ANALYST, xx., 116). To obtain satisfactory results, the potassium xanthate used must be properly prepared. A weighed amount of fused potassium hydroxide is placed with absolute alcohol (2·5 c.c. per gramme potash) in a tightly-stoppered flask, and digested cold, with occasional shaking, until solution is complete, except for the potassium carbonate present. The solution is poured off, cooled in ice-water, and pure carbon bisulphide added gradually with constant stirring, until 1 c.c. has been added for every gramme of potash. The liquid is kept in ice-water till the temperature is reduced to 10° C. The precipitated xanthate is filtered off, washed once with alcohol and twice with ether, and dried at 100° C. A. M.

Estimation of Selenium and Tellurium. **E. Keller.** (*Journ. Amer. Chem. Soc.*, 1900, xxii., 241.)—The method depends upon the fact that selenium may be entirely precipitated from solution in concentrated hydrochloric acid by means of sulphur dioxide, whereas tellurium is precipitated only when the acid is diluted somewhat.

To determine these elements in a sample of commercial copper, 100 grammes are weighed out, 2 to 4 grammes of ferrous sulphate added, and dissolved in 400 c.c. of nitric acid. The nitrous fumes are expelled, the solution is diluted and cooled, and sufficient ammonia is added to dissolve all the copper. The ferric hydroxide, containing the selenium and tellurium, is filtered off, and freed from copper by redissolving and reprecipitating. It is then dissolved in a minimum of dilute hydrochloric acid, the liquid is cooled, and sulphuretted hydrogen is passed to saturation. The sulphides are filtered and washed, and digested with a solution of sodium sulphide, which takes up the sulphides of selenium and tellurium. The solution, after filtering, is acidified with nitric acid and evaporated to dryness, taking care not to lose any selenium dioxide. Two hundred c.c. of strong hydrochloric acid (specific gravity 1·16) are now added, and the liquid is boiled until all the nitro-hydrochloric acid is destroyed and the two elements are reduced to the selenous and tellurous state. After cooling, the solution is filtered through asbestos, and the filter is washed with strong hydrochloric acid. Sulphur dioxide is then passed through the filtrate. After settling, the selenium is collected on a weighed Gooch filter, and washed with strong hydrochloric acid about three times, then washed successively with warm dilute hydrochloric acid, pure water, and alcohol. After drying, it is weighed.

The filtrate containing the tellurium is diluted with an equal bulk of water, boiled for a few minutes, and sulphur dioxide is passed through at the same time. After cooling and settling, the tellurium is filtered, washed, and weighed in the same manner as the selenium. A. M.

The Colorimetric Estimation of Vanadium. **L. Maillard.** (*Bull. Soc. Chim.*, 1900, xxiii., 559-563.)—This method is based upon the reaction described by Barreswill in 1847, in which, on treating a strongly acidified solution of a vanadate with ether saturated with hydrogen peroxide, or with ozonized ether or turpentine, a red coloration due to pervanadic acid is obtained.

For the standard, the author uses a 10 per cent. solution of pure sodium metavanadate, which is diluted as required to 1 : 100 or 1 : 1,000.

Ten c.c. of the neutral solution under examination are acidified with 1 to 5 c.c. of hydrochloric acid in a graduated cylinder, into which are then introduced 3 to 10 c.c. of ether saturated with hydrogen peroxide. On shaking the closed cylinder there is an immediate red coloration, while the supernatant ether remains colourless. The aqueous layer is then diluted to 15 c.c., and the colour matched in the colorimeter with that given by the standard solution of metavanadate under the same conditions.

The reagent is prepared by mixing equal volumes of ether and ordinary commercial hydrogen peroxide (10 volumes), and allowing the flask to stand for some time.

A series of determinations are described to show that the method is accurate.

and that the presence of impurities, such as chlorides, sulphates, nitrates, and phosphates, do not interfere with the results.

The red coloration is perceptible in a solution containing 1 part of vanadic acid in 84,000. C. A. M.

The Preparation of Standard Solutions of Sulphuric Acid. A. Marshall. (*Journ. Soc. Chem. Ind.*, 1899, 4, 1091.)—Pure sulphuric acid is mixed with water so that it contains between 62 and 82 per cent. H_2SO_4. The density is then determined, and the exact strength of the acid is calculated from the formula

$$P = D(85{\cdot}87 + {\cdot}05T - {\cdot}0004t^2) - 69{\cdot}80,$$

where P is the percentage of H_2SO_4 in the acid, and D is the density of the acid at T° C., referred to water at t° C. The density need not be determined beyond the third decimal place, and the temperatures need to be known within half a degree only. The above formula may be used for any temperatures from 0° to 40° C. The error in the percentage of H_2SO_4 found is not greater than $\pm 0{\cdot}1$.

The author also gives tables from which the strength of the acid can be obtained accurately to 0·01 per cent.

The strength of the acid being known, a simple calculation gives the amount which must be weighed out to make the required standard solution. A. M.

REVIEWS.

FLESH FOODS: THEIR CHEMICAL, MICROSCOPICAL, AND BACTERIOLOGICAL EXAMINATION. By C. AINSWORTH MITCHELL, B.A. (Oxon.). London: Charles Griffin and Co. Price 10s. 6d.

THIS work fills a distinct gap in literature, for no book covering the whole of the ground taken here has yet appeared in the English language. It commences with a description of the structure and chemical composition of the muscular and other animal tissues, as well as the blood, a section on the identification of blood-stains being included. The characteristics of the flesh of the various animals usually used for food are described, along with their chemical composition, special attention being given to the chemistry of the fats, this latter being a subject upon which the author, in conjunction with Mr. Otto Hehner, has done much original investigation. The general appearance of healthy and diseased flesh are described, as are also the few chemical tests which have been proposed for the detection of unsound flesh, and the relative digestibility and the heat-values of the various flesh foods conclude the chapter. The next is devoted to the methods for examining animal fats. The preservation of flesh and the composition and examination of preserved flesh products are next dealt with, as also is the examination of tinned foods for decomposition, metallic contamination, etc.; preservation of food by means of chemical antiseptics, the effects of their application and their detection, are described. The composition and analysis of sausages forms the subject of another chapter. The animal proteids and their derivatives are next dealt with, several of the schemes for their classification which have been proposed of late years being

given, also the chemical reactions and physical properties of these bodies, with methods for their determination; the processes of artificial digestion, both by chemical and enzymic agent, being appended. The changes produced in flesh by cooking are next described, as well as the effect of that process on animal parasites, bacteria, toxines, etc. Poisonous flesh, its various causes, and the symptoms of poisoning produced thereby, are next dealt with; a list of the principal ptomaines is given, the detection and isolation of which are reserved for the last chapter in the book. Next comes an account of the animal parasites occurring directly or indirectly in flesh usually used for food, together with their detection; this, which is fully illustrated by figures of the various organisms, forms a novel feature in works of this kind. The effect of cooking and curing on the destruction of these obnoxious organisms is also discussed. Next follows a description of the methods for examining flesh for bacteria, a description of the most important of those which usually occur in flesh foods, the diseases caused by them and their thermal death-points. From the above it will be seen that the scope of the work is an exceedingly wide one, and as a consequence the information it contains must necessarily appear in a much condensed form to bring it within the compass of a small handbook. This has, however, been done so intelligently that the general value of the book is not impaired, and it will be found of great value not only to Medical Officers of Health and others who are engaged in the inspection of food, but also to analysts, to whom samples of unwholesome food are frequently referred. The general get-up of the book is excellent, the figures good, and it possesses a copious index. W. J. S.

LUBRICATION AND LUBRICANTS. By L. ARCHBUTT and R. M. DEELEY. London: C. Griffin and Co. Price 21s.

The whole of the extensive subject covered by the title is treated of in this volume. Starting with the discussion of friction generally, the authors pass to those physical measurements, such as viscosity, which serve to ascertain the quality of lubricants and their suitability for a proposed use. They give a full and accurate account of the numerous experiments which have been carried out by Mr. Beauchamp Tower and others to determine precisely what is the behaviour of a lubricant under different conditions. There is a large chemical section of the book which is as excellently rendered as those who know the chemist of the partnership of authors would expect. It is this section that analysts will naturally most eagerly consult, but they may be assured of good return for their labour if they will also study the more mechanical portions of the book. Oil-testing machines, bearings of various design, and the lubrication of machinery, are the subjects occupying the remainder of the volume. To give a full appreciation of this admirable treatise would require the issue of a pamphlet rather than the summary of a review, and in view of this impossibility it is sufficient to say that those chemists who are accustomed to examine and pronounce upon the quality of lubricants will do well to fortify their knowledge and to check their conclusions by consulting a work which is as full of fact as it is of suggestion. B. B.

THE ANALYST.

DECEMBER, 1900.

PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS.

THE monthly meeting of the Society was held on Wednesday evening, November 7, in the Chemical Society's Rooms, Burlington House. The President (Mr. W. W. Fisher, M.A.), occupied the chair.

The minutes of the previous meeting were read and confirmed.

THE MARGARINE CLAUSE OF THE FOOD AND DRUGS ACT, 1899.

The PRESIDENT then read the following report, presented to the Council by the Committee which was appointed, at the suggestion of the Principal Chemist of the Government Laboratory, to confer with him on the subject of Section 8 of the Sale of Food and Drugs Act, 1899. The object of the conference was to arrive at some kind of agreement as to the manner of estimating the proportion of butter-fat in margarine (which by the section referred to was restricted to 10 per cent.), with a view to avoiding discrepancies which might arise through the employment of different methods by individual analysts. The investigations made in the Government Laboratory, and by the several members of the Committee, had been followed by recommendations as to the method of analysis and the calculation of results, which were fully set out in the report and appendix. The report had been agreed to by Dr. Thorpe and by the Committee as representing the Society of Public Analysts, and it had been incorporated in the recently published official report of the Principal Chemist of the Government Laboratory; so that, as far as the Government Laboratory was concerned, the method recommended stood as the official method for determining the percentage of butter-fat in margarine.

REPORT.

In view of prosecutions which may be raised under this clause, it is desirable that an agreement should be come to among analysts as to the method and the analytical criteria which should be employed to raise a presumption, until the contrary is proved, that the amount of butter-fat in margarine exceeds the statutory limit.

On the suggestion of the Principal Chemist of the Government Laboratory, a Committee of the Council of the Society of Public Analysts was appointed to confer with him on this subject.

After a careful examination of methods of analysis and a series of determinations, made independently, of various samples of commercial margarine of known composition, and of mixtures prepared in the Government Laboratory of margarine with varying but known proportions of butter-fat of different Reichert-Wollny values, it has been agreed to recommend:

(1) That the proportion of butter-fat should be deduced from a determination of the amount of the "volatile acids," as ascertained by the Wollny modification of the Reichert-Meissl process, described below.

(2) That no presumption against the margarine in regard to its content of butter-fat should be raised unless the Reichert-Wollny number as ascertained by the method described exceeds 4.

(3) That the amount of butter-fat in margarine, when it exceeds the legal limit of 10 per cent., as determined by the method prescribed, shall be assumed to be as follows:

Reichert-Wollny Number of the Mixture.	Percentage of Butter-Fat present in the Mixture.
4·0	10
4·3	11
4·6	12
4·9	13
5·2	14
5·5	15
5·9	16
6·2	17
6·5	18
6·8	19
7·1	20

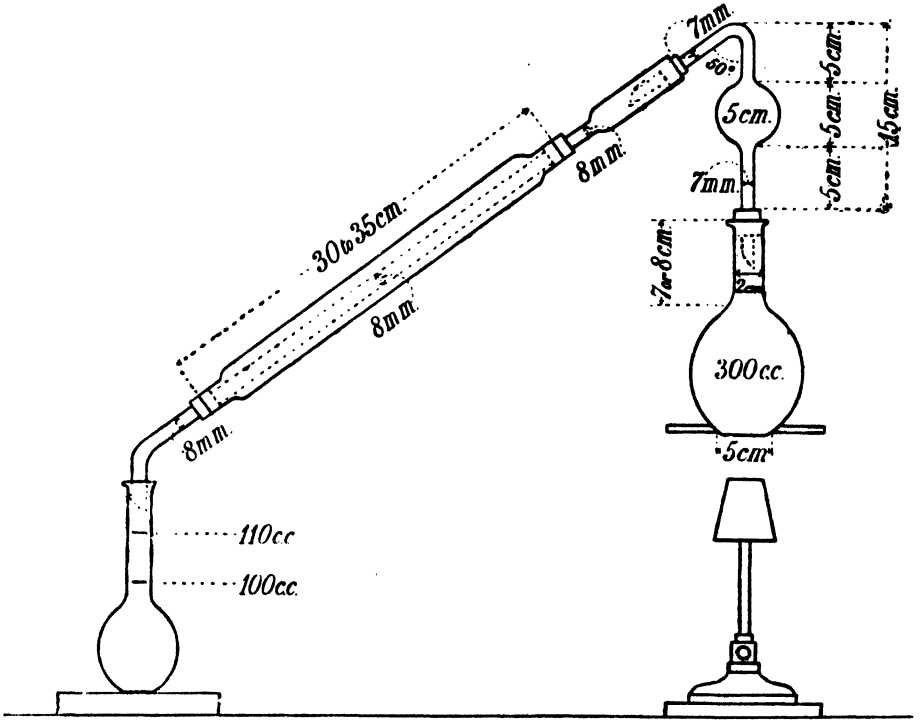
The foregoing table is to be regarded as applicable only to the determination of butter-fat in margarine.

(Signed)
T. E. THORPE,
W. W. FISHER,
A. H. ALLEN,
E. J. BEVAN,
O. HEHNER.

THE REICHERT-WOLLNY METHOD FOR DETERMINATION OF VOLATILE FATTY ACIDS IN MARGARINE AND BUTTER.

Five grammes of the liquid fat are introduced into a 300 c.c. flask, of the form seen in the figure (length of neck 7 to 8 centimetres, width of neck 2 centimetres). Two c.c. of a solution of caustic soda (98 per cent.) in an equal weight of water —preserved from the action of atmospheric carbonic acid—and 10 c.c. of alcohol (about 92 per cent.) are added, and the mixture is heated under a reflux condenser, connected with the flask by a T-piece, for fifteen minutes in a bath containing

boiling water. The alcohol is distilled off by heating the flask on the water-bath for about half an hour, or until the soap is dry. One hundred c.c. of hot water which have been kept boiling for at least ten minutes are added, and the flask heated until the soap is dissolved. Forty c.c. of normal sulphuric acid and three or four fragments of pumice or broken pipe-stems are added, and the flask is at once connected with a condenser by means of a glass tube 7 millimetres wide and 15 centimetres from the top of the cork to the bend. At a distance of 5 centimetres above the cork is a bulb 5 centimetres in diameter. The flask is supported on a circular piece of asbestos 12 centimetres in diameter, having a hole in the centre



5 centimetres in diameter, and is first heated by a very small flame, to fuse the insoluble fatty acids, but the heat must not be sufficient to cause the liquid to boil. The heat is increased, and when fusion is complete 110 c.c. are distilled off into a graduated flask, the distillation lasting about thirty minutes (say from twenty-eight to thirty-two minutes), the distillate is shaken, 100 c.c. filtered off, transferred to a beaker, 0·5 c.c. of phenolphthalein solution (1 gramme in 100 c.c. alcohol) added, and the filtrate titrated with decinormal soda or baryta solution. Precisely the same procedure (with the same reagents), omitting the fat, should be followed, and the amount of decinormal alkali required to neutralize the distillate ascertained. This should not exceed 0·3 c.c. The volume of decinormal solution of alkali used, less the figure obtained by blank experiment, is multiplied by 1·1. The number so obtained is the "Reichert-Wollny Number."

Notes on the Method.—The sample is melted and filtered from curd and water through a dry filter. From the filtrate the 5 grammes of fat for the process are taken. The soda solution is filtered clear from carbonate formed in its preparation, and kept in a special bottle. The Soxhlet spherical condenser is a convenient one for the reflux distillation. This is fixed near the water-bath in which the saponification is to take place, and is connected with the flask by means of a T-piece and indiarubber tubes inclined at an angle of 45°. During the saponification the free limb of the T-piece is directed upwards, and its end closed by a short piece of indiarubber and glass rod. At the end of fifteen minutes this limb is turned downward, and the piece of glass rod replaced by a tube carrying away the alcohol.

One hundred c.c. of hot distilled water are added, and the flask frequently shaken until the soap is dissolved. The Liebig is a convenient form of condenser. One containing a column of water 30 to 35 centimetres in length gives sufficient condensing surface. After shaking the distillate, about 5 c.c. are filtered through a dry paper into a 100 c.c. flask. This serves to wash out the flask. When the 100 c.c. are transferred to a beaker, the flask is not washed out, but the main quantity is neutralized with the standard solution of alkali and returned to the flask, then again transferred to the beaker and the titration completed.

The PRESIDENT then said that some explanation might be given of the adoption of a Reichert-Wollny number of 4 as representing 10 per cent. of butter-fat. Ten per cent. of butter-fat, if the butter had a Reichert-Wollny number of 30, would be represented by a Reichert-Wollny number of 3. The margarine itself, however, must always count for something, and the traces of volatile acids which it would probably contain might raise the number 3 to 3·5 or 3·7; so that, by way of margin to avoid disputes about very small proportions of butter-fat, which, after all, were immaterial, the number 4 was adopted, as being a whole number and more satisfactory than 3·85 or 3·9 would be, and giving the same practical results.

The appendix to the report contained a description of the method of analysis, with a diagram of the apparatus. The apparatus should be constructed in accordance with the measurements given, in order that the results might not be affected by minor differences of procedure.

Dr. RIDEAL said that this report was especially to be welcomed because it marked a new and satisfactory departure in the history of the Society and its relations with the Government Laboratory. The method described was practically the one which he had been in the habit of using for some years past. He would be quite prepared, and he hoped that every public analyst would be prepared, to adhere rigidly to all the details of the method as now laid down, because he believed that only by so doing could uniformity of results be secured; and from personal experience he was sure that its employment would lead to the avoidance of many disputes. He had much pleasure in moving that the Society formally adopt this method as an official method.

Major CASSAL seconded the motion. He agreed that the action which had been taken by the Society in this matter was one of considerable importance, and it was desirable that action of a similar kind should be taken in regard to other products.

The adoption of an official process was necessarily a matter of compromise under certain circumstances, and it would appear that the labours of the Committee had resulted in the adoption of a process which was satisfactory to both parties concerned. It was obviously important that in this, as in other matters, the Society should be in agreement with the Somerset House analysts, who had met the Society in a fair and proper spirit, which he had no doubt whatever would also be exhibited by them in the future in regard to any other questions that might arise.

Mr. EMBREY supported the motion, on the ground that it was quite as important that public analysts should agree amongst themselves as that they should agree with the Government chemists. There were, he thought, points in the method recommended which were open to criticism, but he nevertheless would be prepared to adhère to it, seeing that it had been agreed upon.

The motion, on being put to the meeting by the President, was carried unanimously.

A certificate of proposal for election to membership in favour of Mr. John Stewart Remington was read for the second time. Certificates in favour of Messrs. Percy Henry Carpenter, 46, Streathbourne Road, Upper Tooting, S.W., assistant to Mr. Alfred C. Chapman; and Samuel Russell Trotman, M.A., F.I.C., 1, Waverley Street, Nottingham, Public Analyst for the City of Nottingham, were read for the first time.

The following papers were read: "The Determination of the Available Brewing Extract of Malt," by Lawrence Briant; and "Notes on certain B. P. Tests," by C. G. Moor, M.A., and Martin Priest.

A NEW COLOUR REACTION FOR DISTINGUISHING BETWEEN CERTAIN ISOMERIC ALLYL AND PROPENYL PHENOLS.

BY ALFRED C. CHAPMAN, F.I.C.

(*Read at the Meeting, June* 6, 1900.)

MANY of the odoriferous constituents of essential oils are now known to consist of phenols or their simple derivatives, and some of these are of considerable technical importance, chiefly owing to the fact that they serve as the starting-points in the manufacture of certain well-known artificial perfumes.

Thus, eugenol and safrol serve respectively for the preparation of the important perfumes vanillin and heliotropin.

Many of these phenolic substances may be grouped in pairs, the one isomer containing the allyl grouping $CH_2 . CH : CH_2$, whilst the other has instead the propenyl group, $CH : CH . CH_3$, and the conversion of the former into the latter can, as a rule, be effected without difficulty.

In some cases, as, for instance, eugenol and safrol and their isomers, the general properties of the pair of compounds are so much alike that a simple colour reaction capable of distinguishing clearly and easily between them is of sufficient interest, I think, to warrant me in bringing it before the notice of the Society.

The reaction in question is brought about by simply dissolving 1 c.c. of the

phenol in 5 c.c. of acetic anhydride, and then adding (*a*) a fragment of fused zinc chloride or (*b*) 1 drop of concentrated sulphuric acid.

Experiments were made with the six substances, eugenol and iso-eugenol, safrol and iso-safrol, and estragol and anethol, and the colour changes recorded below were observed :

	Eugenol.	Iso-eugenol.
H_2SO_4.	Brown at first, becoming quickly purple, and finally wine-red.	A rose pink, quickly changing to a light brown.
$ZnCl_2$.	Pale yellow, the colour disappearing on standing.	Bright rose-pink coloration.
	Safrol.	**Iso-safrol.**
H_2SO_4.	A bright emerald-green colour, becoming brownish-green and finally brownish.	A faint transient pink, becoming reddish on standing.
$ZnCl_2$.	Pale blue, becoming less intense on standing, and finally light brown.	Pink, becoming brownish-pink and finally brown.
	Estragol.	**Anethol.**
H_2SO_4.	Purple, turning to indigo blue, and then to bluish purple.	No colour at first. After a short time a yellowish tinge.
$ZnCl_2$.	Blue-violet colour, which becomes deep mauve and finally brownish.	A pale yellow colour appearing slowly, which deepens on standing, and finally becomes brick-red.

Seeing that colour reactions are so largely influenced by the presence of impurity in the compounds to which they apply, I may add that no pains were spared to obtain the above substances in as pure a condition as possible.

Some of them (eugenol, iso-eugenol and iso-safrol) were prepared in my laboratory, whilst the remaining three were procured from Kahlbaum of Berlin, and purified by me.

In all cases the chief physical constants were carefully determined and compared with those given by the most reliable authorities, and when possible crystalline derivatives were prepared from them and identified.

There can, therefore, I think, be no reasonable doubt that all were in as high a degree of purity as it is possible to obtain them.

Discussion.

Dr. Lewkowitsch said that, in the case of the so-called colophony test, a more distinct reaction was obtained with sulphuric acid of specific gravity 1·53 than with an acid of greater strength ; and perhaps in the case of the test now described a somewhat weaker acid would give a more strongly developed coloration, as seemed to be shown by the zinc chloride.

Mr. A. Marshall inquired whether any other dehydrating agents had been tried.

Mr. Chapman said that the treatment of these hydroxy-compounds in the manner described was the ordinary method of preparing acetyl derivatives, and his attention had been quite accidentally directed to the reaction in that way. The investigation had not been carried any further, but he thought that the test as it stood would probably suffice for ordinary purposes.

DETECTION OF MAIZE IN WHEATEN FLOUR.

By G. Embrey.

(*Read at the Meeting, June* 6, 1900.)

During the past three or four years English millers have been seriously affected by the introduction of foreign wheaten flour, containing from 10 to 30 per cent. of maize flour; and yet very few prosecutions of dealers selling such have taken place.

Many fancy articles sold, as "Self-raising Flour" and "Pastry Flour," have also been found to contain from 10 to 20 per cent. of maize.

I have examined the published methods for detecting and estimating this substance, but find the results are not so constant as is desirable for proceedings under the Food and Drugs Act.

If we compare the flours separately as regards their chemical composition, we fail to discover variations sufficiently wide to enable us to estimate one in the presence of the other.

At one time I thought it possible to make use of the difference in the amount of fat found in the flours; but although the whole-grain of maize contains nearly 5 per cent. of fat, the samples of white maize flour on the market contain only 2·84 per cent. of fat, while wheat flour contains from 1·2 to 2 per cent. of fat.

There is some difference in the amount and in the character of the mineral matter. Thus, while maize contains 0·714 per cent. ash, nearly 60 per cent. of which is soluble in water, and which has 38·65 per cent. phosphates, calculated as tricalcium phosphate, wheat flour has usually only 0·42 per cent. of ash, 71 per cent. of which is soluble in water, 22·42 per cent. of the ash being phosphates; so the examination of the ash may be used to confirm the other methods of estimation.

I believe the usual method of detecting maize in presence of other flours is by means of the microscope; and the mode described by Allen, aided by the excellent drawing (facing page 411, "Commercial Organic Analysis," vol. i., third edition), is all that can be desired.

In a paper on "The Use of Maize as an Adulterant in Oatmeal," by J. White, F.I.C. (Analyst, vol. xx., p. 30), the author gives some excellent advice as to the microscopical examination of flours containing maize.

Still, I have been unable to obtain concordant results in quantitative estimations when using only the microscope and its appliances.

In vol. xxiv., p. 150, of the Analyst will be found an abstract from an article by K. Bauman concerning this subject, and a method both for detection and estimation of maize is given; but while his method of detecting it is simple and effective, that for its estimation does not work well in my hands. However, it is an extension of this method which I bring before the society to-night.

Bauman points out that a 1·8 per cent. solution of potassium hydroxide will under certain conditions gelatinize wheat starch, but leave maize starch unaffected. This I find to be the case, and taking advantage of this, have worked out the following modification and extension:

1. It is necessary to provide samples of pure wheaten and pure maize flours.

2. The following solutions are required:

(1)	Potassium hydroxide	18 grammes.
	(Make up to a litre with distilled water.)	
(2)	Iodine	0·25 gramme.
	Potassium iodide	1 gramme.
	Distilled water to	250 c.c.
(3)	Hydrochloric acid, specific gravity 1·16	50 c.c.
	Distilled water to	100 c.c.

Process.—Weigh out the following:

(*a*)	0·2	gramme of the sample under examination.								
(*b*)	0·18	gramme	wheat flour, and	0·02	gramme	maize flour	= 10	%	of	maize flour.
(*c*)	0·17	,,	,,	,,	0·03	,,	,,	= 15	,,	,,
(*d*)	0·16	,,	,,	,,	0·04	,,	,,	= 20	,,	,,
(*e*)	0·15	,,	,,	,,	0·05	,,	,,	= 25	,,	,,
(*f*)	0·14	,,	,,	,,	0·06	,,	,,	= 30	,,	,,

Place each of the above in test-tubes (15 centimetres × 2 centimetres) fitted with paraffined corks, then add to each 20 c.c. of the potassium hydroxide solution; shake uniformly for three minutes; add 12 drops of the hydrochloric acid; place in centrifugal machine revolving 600 times per minute; remove 1 c.c. of the liquid, avoiding carefully the residue at the bottom of the tube; make up to 50 c.c. in Nessler glass; add two drops of the hydrochloric acid, then 1 c.c. of the iodine solution; stir and compare tints; this will give the amount of maize flour present in the sample to within 5 per cent. only, which is not near enough for our purpose.

But if we take 10 c.c. of the clear liquid from each test-tube, add 1 c.c. of dilute sulphuric acid (1 : 7), and boil for two hours (thus entirely converting the starch into sugar), then neutralize, make up to 50 c.c., well mix, and place in a burette, and run into the following mixture, which is kept boiling:

Gerrard's solution*	10 c.c.
Fehling's solution	2 c.c.

until the colour is discharged, the made-up specimen, requiring the same amount to discharge the colour as the sample under examination, will give the percentage.

Discussion.

Mr. Bevan said that he had recently tried, with satisfactory results, a qualitative method for the detection of the presence of maize in wheaten flour which had been communicated to him by Mr. A. C. Wilson, of Stockton-on-Tees. In this the flour, or starchy compound, is mixed with clove oil, and examined under a $\frac{1}{4}$- or $\frac{1}{8}$-inch objective, when the hilum of maize appears as a black dot or star; the wheat or other starches are practically invisible.

* On p. 75 of Allen's "Chemistry of Urine," these directions are given for Gerrard's process:

"Ten c.c. of freshly-prepared Fehling's solution, or 5 c.c. of each of the constituent solutions, both accurately measured, should be placed in a porcelain dish, 40 c.c. of water then added, and the liquid heated to boiling. A solution of potassium cyanide, of about 5 per cent. strength, is gradually added from a burette or pipette until the deep blue colour of the liquid is nearly destroyed. The addition of the cyanide is continued very cautiously drop by drop, the liquid being kept constantly boiling and well stirred until the blue colour just disappears, or only a very slight tinge of blue remains. Excess of cyanide solution must be carefully avoided, so that it is safer to discontinue its addition when the blue colour is still faintly perceptible."

Mr. JOHN WHITE said that a qualitative test—which might also be made quantitative—for the detection, and, if necessary, the determination of maize in wheaten flour, was communicated to him some three or four years previously by Mr. E. W. T. Jones, and he had found it to answer all practical requirements. After the sample had been thoroughly mixed in a mortar, 1 gramme of it was placed in a test-tube, 10 c.c. of distilled water added, and the mixture well shaken; 15 c.c. of a 1 per cent. solution of sodium hydroxide was then added, and the whole again shaken. After standing for not less than three or four hours, nearly the whole of the wheat starch was gelatinized, as in the case of the treatment with caustic potash; and, if maize was present, a drop of the emulsion, examined under the microscope with a ¼-inch objective and an A or B eyepiece, showed the granules of maize very distinctly. Wheat of a certain special class appeared to resist the action of the alkali for a considerable time, but by polarized light this was easily distinguishable from the granules of maize. In the case of a sample examined jointly, the conclusions independently arrived at by Mr. Jones and himself had been in very close agreement.

ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

FOODS AND DRUGS ANALYSIS.

Acidity of Milk. Vieth and Siegfold. (*Milch Zeit.*, 1900, xxix., 593; through *Chem. Zeit. Rep.*, 1900, 283.)—The authors publish two sets of observations, carried out at different times and places, to ascertain whether the acidity of milk, as produced under different conditions, is always approximately constant, and whether milk can be delivered to a dairy after a long journey in the hot season of the year perfectly sweet. The figures show that a high acidity is found not only when the milk is going sour, but also when an early souring can hardly be expected; so that the acidity must be considered natural. It is also evident that even in hot summer weather delivery of a mixed daily milk in good condition is quite possible, even if part of the bulk was undoubtedly sour. The natural acidity of milk is in no way constant in amount, but varies within sensible proportions. F. H. L.

Estimation of Fat in Condensed Milk. A. E. Leach. (*Journ. Amer. Chem. Soc.*, vol. xxii. [9], pp. 589-591.)—In the case of condensed milk containing added cane-sugar, the sample is well stirred to render it homogeneous, and 40 grammes are weighed out, washed into a 100 c.c. flask and made up to the mark with water. Twenty-five c.c. are placed in a test-bottle, which is then nearly filled with water and 4 c.c. of copper sulphate solution, of the strength of Fehling's copper solution, the whole being well shaken up and placed in a Babcock centrifuge, preferably driven by electricity, since heat cakes the precipitate and makes it harder to wash. When the separation is complete, the supernatant liquid is drawn off by a pipette, through a

filtering wisp of cotton, the precipitate is washed thoroughly in the machine; and, finally, enough water is added to amount approximately to the normal volume of 17·6 c.c. usually employed for the Babcock test, the subsequent stages of which are then carried out in the ordinary manner. C. S.

Detection of Coal-Tar Dyes in Fruit Products. A. L. Winton. (*Journ. Amer. Chem. Soc.*, vol. xxii. [9], pp. 582-588.)—The author recommends Arata's wool test, applied by boiling 100 c.c. of the liquid under examination for ten minutes with 10 c.c. of 10 per cent. potassium bisulphate solution, and a piece of white wool previously boiled in dilute sodium hydroxide and washed, since the natural colouring matters of fruit impart at most a pink or brown coloration which turns green in presence of ammonia, and is not restored on washing with water; whereas coal-tar dyes—the azo-group particularly—produce colorations either unaffected by ammonia or restorable by washing. Care should, however, be taken to ascertain whether the wool is really dyed, or merely coated with the colouring matter; chlorophyll, for example, and colouring preparations of vegetable origin, since they are merely deposited on the surface, are removed by friction or washing. It is better to apply the subsequent identification tests to the dyed wool rather than to the dye extracted therefrom. The amyl alcohol (alkaline or acid solution) confirmatory tests for fuchsine may be afterwards employed, as well as Gerard's mercuric acetate method and Cazeneuve's mercuric oxide test. The two last are, however, unsuitable for jellies, though useful for fruit juices and syrups. In reports it is considered better to avoid attempts at particularizing the dye-stuff present, beyond indicating the colour, *e.g.*, 'red' or 'orange coal-tar dye.' C. S.

Determination of Vanillin. J. Hanus. (*Zeit. für Untersuch. der Nahr. und Genussmittel*, 1900, iii., 531-537.)—The experiments described were carried out with the object of devising a method for the determination of vanillin by the production of a hydrazone of suitable character by the addition of one of the hydrazines. Of those experimented with only the β-naphthylhydrazine hydrochloride and the *p*-bromphenylhydrazine were found to satisfy the requirements.

β-Naphthylhydrazine hydrochloride ($C_{10}H_7.NH.NH_2.HCl$).—The experiments were carried out as follows: The β-naphthylhydrazine solution was added to the vanillin solution in such proportion that 2 to 3 parts of hydrazine were present for every part of vanillin. The mixture was allowed to stand for five hours, after which it was filtered through a dried and weighed filter, and washed with hot water until the washings no longer exercised a reducing action upon silver nitrate. The precipitate was then dried at 90° C., and weighed.

To effect complete separation it is necessary that the mixture be allowed to stand for some time, though, on the other hand, standing too long may lead to an error in the opposite direction through the production of oxidation products of hydrazine, which cannot be removed by washing. Standing for five hours appears to give the best results.

p-Bromphenylhydrazine ($C_6H_4Br.NH.NH_2$).—Two to three parts of hydrazine

were used for each part of vanillin. The mixture was allowed to stand for four or five hours, after which it was filtered and washed as before. Finally the precipitate was dried at 100° C., and weighed. The results by both processes were satisfactory.

This method is applicable in all cases where an aqueous solution of the vanillin can be prepared, and it has the advantage over Welman's method (titration with alcoholic potash) of being unaffected by the presence of vanillic acid. H. H. B. S.

Researches on Cayenne Pepper. G. Gregor. (*Zeit. für Untersuch. der Nahr. und Genussmittel*, 1900, iii., 460-471.)

1. *On the Assimilability of Metallic Salts by the Cayenne Pepper Plant.*—In a paper contributed to the *Chem. Zeit.* (1899, xxiii., 433) Jonscher called attention to the fact that 0·91 per cent. of barium oxide in the form of readily soluble salt had been found in a sample of ground cayenne pepper. The publication of this paper, and its subsequent discussion in the columns of the journal, led the author to undertake the present investigation. The experiments were carried out as follows: Cayenne pepper plants were cultivated in wooden boxes filled with ordinary garden soil. In one case the soil was used alone; in two other cases barium sulphate and calcium sulphate respectively were mixed with it, whilst in the remainder the soil was watered respectively with solutions of barium chloride, calcium chloride, lead chloride, and copper sulphate. The experiments were commenced in July, and the ripe fruit was collected in October. After drying in the usual way, the percentage of ash in the fruit from each experiment was determined and examined for the particular metals experimented with. From the results obtained, the author concludes that neither barium nor lead is capable of being assimilated by the cayenne pepper plant, and that consequently the presence of these metals in the ash of the fruit must be taken as a proof of admixture.

2. *On the Percentage of Ash in different Sorts of Cayenne Pepper.*—The fruit grown by the author for the purpose of the preceding investigation was found to yield from 9·74 to 13·37 per cent. of ash, the latter result being obtained in the case of fruit grown upon soil containing calcium sulphate watered with solution of sodium chloride. These figures are not only much in excess of those obtained by other investigators, but are also above the maximum fixed by the German Union for establishing standard methods for the examination of food products, viz., 6·5 per cent., with 1 per cent. insoluble in hydrochloric acid, as well as above the maximum proposed by Swiss chemists, viz., 5 per cent., with 2·5 per cent. insoluble. In order, therefore, to further investigate the subject, the author collected local specimens of normally grown fruits for examination, and with these he found from 7·12 to 10·03 per cent. ash, and from 0·22 to 0·34 per cent. of this soluble in hydrochloric acid.

Further, the results of the examination of twenty-five commercial samples of ground cayenne pepper from various sources gave from 5·71 to 8·92 per cent. of ash, of which from 0·17 to 2·00 per cent. was soluble in hydrochloric acid.

The author remarks that the high percentage of ash insoluble in hydrochloric acid found in some of the samples examined must be due either to accidental

impurities, or to admixture with some foreign substance. Further examination of this part of the ash showed that the excess consisted in some cases of barium sulphate and in others of lead oxide. The proportion of barium sulphate was, in the author's opinion, too insignificant to admit of the supposition that it was purposely added to increase the weight. He considers it more probably due to the practice of artificially colouring the ground pepper. A colouring-matter sold for this purpose under the name of "chrome red" was found to consist of chrysaurein mixed with 60 per cent. of barium sulphate. H. H. B. S.

TOXICOLOGICAL ANALYSIS.

Detection of Sulphonal, Trional, and Tetronal in Cases of Poisoning. D. Vitali. (*Boll. Chim. Farm.*, 1900, xxxix., 461; through *Chem. Zeit. Rep.*, 1900, 243.)—The animal matters submitted for examination are warmed in a porcelain basin three times with twice their volume of 90 per cent. alcohol; after cooling, the extracts are filtered, and the solvent distilled off. The residual aqueous liquid is warmed, filtered, made alkaline with potassium hydroxide, and exhausted three times with an equal volume of ether. (Potash has no effect on sulphonal; it is only employed to prevent colouring matter passing into the ether.) The ether is driven off, leaving the sulphonal behind. It can then be identified by the reactions described by Wefers-Bettinck, Schwarz, Ritsert, and Vulpius, or by Vitali's own tests as follows: (1) When sulphonal is fused with three times its weight of potash, a garlic-like odour is evolved; the mass becomes yellow, then red, and scarlet when cold. If the melt is extracted with water, a turbid, bluish liquid is obtained, and in the filtrate polysulphides can be detected by means of sodium nitroprusside, thiosulphate by acidification. If the mixture of sulphonal with potash is heated to the melting-point of glass, the red colour changes to a blue, owing to the formation of a body analogous to ultramarine. (2) When sulphonal is warmed with a little metallic sodium, sodium sulphide is produced, as may be recognised by treatment with water and nitroprusside. (3) If sulphonal is heated with twice its weight of potassium periodate (only small quantities should be used), a slight explosion occurs, and potassium sulphate is produced. This test succeeds with 1 milligramme of substance.

Sulphonal resists putrefaction, and it can therefore be recovered without loss by the above process from animal remains in a state of decomposition. The same method of isolation, and the same tests, are available in the case of trional or tetronal. They can be distinguished one from another by their different melting-points and by the different appearance under the microscope of the crystals obtained by the spontaneous evaporation of their aqueous or ethereal solutions. Sulphonal and trional are only eliminated in their original condition through the urine in small quantities. They may be detected therein by concentrating the urine on a water-bath to the consistency of an "extract," treating the residue with boiling alcohol, filtering, distilling off the spirit, again filtering the aqueous liquid, boiling with potash to destroy urea, and finally shaking out with ether.

(*Cf.* ANALYST, 1899, xxxiv., 129.) F. H. L.

ORGANIC ANALYSIS.

The Carbohydrates of Irish Moss (Carrageen). J. Sebor. (*Oester. Chem. Zeit.*, 1900, iii., 441.)—The mucilage yielded on decoction of Irish moss contains a complex carbohydrate yielding on hydrolysis galactose, dextrose, and lævulose, also a small quantity of a pentosan, probably xylan. The sugars, however, are not present in the same proportion as in raffinose. Whether the moss contains a mixture of the three corresponding pentosans or a carbohydrate yielding the three sugars cannot be ascertained; but as the mucilage has an organic structure, is insoluble in water, easily yields colloidal solutions like starch paste, and is readily converted into dextrin-like substances, it would appear to be a vegetable reserve-stuff of great molecular complexity similar to starch. F. H. L.

Cedar-nut Oil. L. von Schmoelling. (*Chem. Zeit.*, 1900, xxiv., 815.)—This oil is obtained from the nuts of the Siberian cedar, or stone pine (*Pinus cembra*), a tree which forms vast forests in the southern districts of that country. It has a golden-yellow colour and a very pleasant, mild taste, though somewhat rancid. It is used in Siberia as a food. The oil is scarcely soluble in cold alcohol, carbon bisulphide or benzene; but in cold petroleum spirit, chloroform, acetone and amyl alcohol it is soluble in all proportions. The three first liquids dissolve it on warming. Mixed with an equal volume of fuming nitric acid, it gives a dark red colour, which is permanent for more than twenty-four hours. Ten drops of oil and 2 drops of strong sulphuric acid show a dark-brown ring with black streaks in the oil; with 1·53 sulphuric acid it exhibits a yellowish-brown colour. The constants of cedar-nut oil are as follows:

	Oil.	Fatty Acids.
Specific gravity	0·930	
Hehner number	91·97	
Saponification value	191·8	
Iodine value (Waller)	159·2	161·3
Acid value	3·25	193·0
Glycerin (esters × 0·0547)	10·31 per cent.	
Volatile fatty acids	3·77 ,,	
Free fatty acids	1·6 ,,	
Total fatty acids	95·74 ,,	
Mean molecular weight	280	290
Acetyl number (after six days' standing) ...	—	81·9
Liquid fatty acids (Muter and De Koningh)	—	87·0 per cent.
Unsaponifiable matter (Fahrion)	1·3 per cent.	
Solidifying point	—	11·3
Iodine value of liquid acids	—	184·0
Temperature reaction (Maumené-Archbutt)...	98°	

Kryloff examined a sample of this oil in 1898, and obtained: Hehner number, 93·33; saponification value, 191·8; iodine value, 149·5 to 150·5; acid value, 1·09; and volatile fatty acids, 2·0 per cent. Among the constituents of the oil palmitic acid

can be recognised, while the liquid acids consist mainly of linolic acid, with some oleic and very little linolenic acid.

On heating the oil with 5 per cent. of manganese borate for four hours to 140° or 150° a "varnish" is produced, which dries on glass in double the time taken by linseed varnish similarly prepared; the product is very viscid, and resembles a blown oil. This last characteristic, coupled with the high price of the original oil, would prevent it from being used in the manufacture of varnish. F. H. L.

Testing for Vegetable Oils by a Modified Welmann's Test. T. Geuther. (*Zeits. öffentl. Chem.*, 1900, vi., 328; through *Chem. Zeit. Rep.*, 1900, 283.)—Finding that the commercial 10 per cent. solution of phosphomolybdic acid is unsuited for analytical purposes, the author proceeds as follows: 5 grammes of pure sodium phosphomolybdate are mixed with 25 c.c. of water and 30 c.c. of pure 1·39 nitric acid, shaking till solution is complete. A test-tube is tared, and into it are weighed 5 grammes of the lard to be examined, after it has been melted, mixed together, and carefully filtered. Three grammes of the purest chloroform are next weighed into the tube, and finally 20 drops of the reagent are added from a pipette. After thorough agitation, the tube is set aside, and the colour which develops within two minutes is observed. In presence of only 5 per cent. of an unrefined and unbleached oil, a distinct dark green tint appears; while pure, fresh, filtered lard gives only a yellow colour. Any green colour that develops after the two minutes may be neglected.

F. H. L.

Action of Sodium Ethoxide on Fats—Volumetric Estimation of Water in Alcohol—Determination of Glycerol in Fats. H. Bull. (*Chem. Zeit.*, 1900, xxiv., 814, 845.)—The process which the author has already described (ANALYST, 1900, xxv., 126) for estimating the unsaturated fatty acids in train oil depends on the assumption that the neutral glycerol esters in those oils are saponified by sodium ethoxide. Such, however, is not the case unless the alcohol in which the original metallic sodium was dissolved contained at least 4 per cent. of water. This quantity of water not only causes complete saponification, but also raises the solubility in ether of the sodium salts of the train-oil unsaturated acids, while an increase in the amount of alcohol present behaves in a similar manner. Bull appends to his present article a table giving the unsaponifiable matter and the fatty acids with soluble sodium salts as redetermined after eighteen months' storage in half-filled bottles on the same oils previously examined (*loc. cit.*), the proportions of fatty acids being now considerably lower, owing, no doubt, to the action of air and light on the samples.

When sodium ethoxide is shaken with a fatty (*e.g.*, train) oil not containing any free acid, an emulsion is first produced, which quickly disappears, leaving a clear solution that deposits fine crystals of mono-sodium glyceroxide according to the equation (F being a fatty acid radicle):

$$C_3H_5.(OF)_3 + C_2H_5.ONa + 2C_2H_5.OH = C_3H_5.(OH)_2.ONa + 3C_2H_5.OF.$$

This reaction occurs almost instantaneously at ordinary temperatures, and

without the production of a sensible amount of heat; it proceeds between an equal number of molecules of fat and ethoxide, instead of between 1 molecule of fat and 3 of sodium hydroxide, as takes place during saponification.

Estimation of Water in Alcohol.—If, then, a neutral fat is boiled with a solution of sodium ethoxide in absolute alcohol, no saponification occurs; but in presence of water a certain quantity of ethoxide is decomposed into alcohol and hydroxide (which reacts with the fat), thus forming a means of calculating the moisture. Normal ethoxide is required, which is standardized by gently warming 10 c.c. with 5 c.c. of a fatty oil as free as possible from uncombined acids (medicinal cod-liver oil, for example) in a perfectly dry flask fitted with an upright condenser. The liquid is cautiously heated for thirty minutes, then allowed to boil for fifteen minutes; finally cooled, diluted with about 20 c.c. of neutral ether, and titrated with semi-normal hydrochloric acid and phenolphthalein. Repeating the process with an alcohol containing some moisture, less standard acid is used in the titration, and the difference calculated into H_2O. Various examples of the method are quoted which show satisfactory agreement among themselves, and which also indicate accurately the strength of the alcohol employed. It is clear that the same process is available for the valuation of other alcohols than ethylic. By altering the conditions the reaction may be utilized to prepare strictly absolute ethyl alcohol. In a sample dried over quicklime, the proportion of water still remaining is determined as above; the necessary amount of metallic sodium or sodium ethoxide and an excess of a neutral fat are added to the bulk, the whole is heated for a short time under an inverted condenser, and the alcohol is distilled off on the water-bath, using diminished pressure towards the end of the operation.

Estimation of Glycerol in Fats.—In a vessel graduated at the 50 c.c. point, and provided with a stopper, 3 grammes of fat and 3 c.c. of 2N sodium ethoxide are well shaken together and heated to 70° C. for thirty minutes on the water-bath. After cooling for half an hour in the air, 25 c.c. of dry ether are added, the whole is agitated, and diluted with more ether to the mark. In three hours' time the sodium glyceroxide, completely thrown out of solution by the ether, has settled to the bottom, and the supernatant liquid is perfectly clear. Twenty-five c.c. thereof are pipetted off, diluted with 10 c.c. of alcohol, and titrated with decinormal hydrochloric acid and phenolphthalein, overlooking the brown tint due to the presence of oxy-acids. The residual 25 c.c. in the tube are next titrated similarly, except that 5 c.c. of semi-normal HCl should be run in at once. From the difference between the volumes of decinormal acid absorbed, the amount of glycerol may be calculated by using the factor 0·0092. If the same pipette is always used, the results should be 0·06 per cent. too low, no allowance being made for the volume of the precipitated glyceroxide. The chief difficulty met with in the process is to ascertain the end-point of titration owing to the brown colour of the liquid; possibly some other indicator would be better than phenolphthalein. Four tests of a whale-oil (acid number, 3·6, saponification number, 188·3) gave 9·69, 9·54, 9·57, and 9·5 per cent. of glycerol; the first result being recognised as about 0·1 per cent. too high, due to adherence of the solution to the pipette.

The author has not succeeded in determining the amount of glycerol in com-

mercial glycerin by this process. Addition of ether causes a precipitation of sodium oxide as well as of the glyceroxide; in this respect benzene is a better precipitant to employ. The water in glycerin is capable of estimation as above; but the practical difficulties of the method render it of little interest. It should be noted that sodium glyceroxide behaves like the ethoxide: it saponifies only in presence of water, and quite slowly, as it is somewhat insoluble in spirit. This fact is important, since such conditions may arise when fats are being hydrolysed in alcoholic liquids of great strength. F. H. L.

Detection of Bile-Pigments. Triollet. (*Bull. Sciences Pharmacologiques*, July, 1900; through *Chem. Zeit. Rep.*, 1900, 284.)—This process is a further modification of that recommended by Gurdin and modified by Jolles. Fifty c.c. of urine are treated with an excess (40 or 50 grammes) of ammonium sulphate, and filtered through cotton-wool, which retains all the precipitated pigments. The wool is extracted with hot chloroform, which dissolves bilirubin and bilifuscin; the solution is evaporated to dryness. The wool is next extracted with hot alcohol, which dissolves biliverdin and biliprasin; the solution is also evaporated. The two residues are taken up in boiling water, yielding a faintly-coloured solution free from blood, albumin, pus, and urea. The liquid is then cautiously floated on to nitric acid containing nitrous acid. At the junction of the liquids two coloured zones appear—one bright red, the other yellow. In ten minutes a green ring appears between the two former, and five minutes later a fine blue colour will be seen between the violet-red and the green. Afterwards the tints become violet-red, blue, green, yellow, and grow in intensity to a maximum in half an hour. After two hours there is a blue ring between two yellow ones, and in five hours only a uniform yellow tint is left.
F. H. L.

Valuation of Glue. A. Heinemann. (*Chem. Zeit.*, 1900, xxiv., 871.)—The author doubts whether the processes hitherto suggested for the examination of glue are of much industrial use. Determination of the viscosity of a solution of known strength may indicate the value of the material if it be required to manufacture "compositions," "hectograph surfaces," etc.; but it does not appear to throw any light on the real adhesive power of the glue. Similarly estimation of water, fat, and ash may be important; but there is no evidence that the binding power is in proportion to the amount to true gelatin in the sample. Heinemann suggests a process which depends on ascertaining the minimum strength of a solution that exhibits adhesive power. Strips of strong, smooth paper $4 \times 3{\cdot}5$ inches are cut up; one of these is brushed over with a (say) 1 per cent. solution of the glue to be examined for a length of 2·5 inches, another dry strip is laid over it, and the whole is pressed together under a 5-pound weight for exactly 1 minute, or if the atmosphere be very damp, for 1½ or 2 minutes. The papers are then lifted, and it is noticed whether the joint will carry a weight. At or about the "critical" degree of dilution, the joint

will either support a fairly heavy weight, even 30 pounds, or nothing at all. By simultaneous comparison with a solution prepared from a standard sample of glue, altering the strength of the experimental solution as may be necessary, results can be obtained which should indicate the adhesive power and therefore the commercial value of the glue. F. H. L.

[Presumably the second strip is laid on the first so as to overlap only for just over the 2½ inches of painted surface, thus leaving at either end a length of 1½ inches of single paper whereby to clip the test piece. F. H. L.]

On Certain Peculiarities in the Urine of Vegetarians. J. H. Long. (*Journ. Amer. Chem. Soc.*, vol. xxii. [9], pp. 592-595.)—In the experiments detailed the reducing power of the urine was apparently about 20 per cent. above the average, but the difference was mainly due to higher concentration, the average daily excretion (874 c.c.) being below the normal. The proportion of creatinin was unusually low, the uric-acid creatinin ratio being 1 : 1·2 instead of 1 : 2, and is probably attributable to low food-consumption and metabolism.

The distribution of the reducing power (total, 6·888 grammes of cupric oxide per 1,000 c.c. of urine) is interesting, 2·373 grammes being allocated, on the basis of previous experience, to uric acid and creatinin, leaving 4·515 grammes of cupric oxide as the amount corresponding to the carbohydrates or similar bodies present. Hence it is considered that a purely vegetable diet, wherein fats and carbohydrates predominate, favours the increase of non-nitrogenous (probably carbohydrate) substances in the urine. C. S.

A Limiting Standard of Acidity for Moorland Waters. W. Ackroyd. (*Chem. News*, vol. lxxxii., pp. 162, 163.)—Plumbism, due to the solvent action of moorland water on lead piping, does not, in the author's experience, occur when the acidity of the water is lower than the equivalent of 0·5 part of sulphuric acid per 100,000 of water, and he therefore proposes this limit should be adopted as a tentative standard of acidity for such waters when the same is estimated by $\frac{1}{100}$ normal alkali in presence of phenolphthalein as indicator. It is stated that the range of acidity in nine samples of water, not above suspicion in respect of causing plumbism, was 0·53 to 0·91 (average 0·63), whilst in sixty-one samples from places where plumbism is unknown the acidity ranges from 0·20 to 0·41, the average being 0·27. C. S.

INORGANIC ANALYSIS.

Influence of Silica upon Silver Assay. L. Strauss. (*Proc. Amer. Inst. Mining Eng., Canadian Meeting*, 1900; through *Chem. Zeit. Rep.*, 1900, 275.)—When silver is assayed in presence of silica, some of the metal passes into the slag, causing loss; and experiments have shown that addition of ferrous sulphide to the

mixture renders the loss almost constant. In order to ascertain the precise action of the silica, Strauss has assayed various mixtures of silver (0·99 fine) and lead with pure ores, such as pyrites, zinc blende, lead glance, etc., sulphur and silica at temperatures of 900° to 1,000° C., keeping the amount of lead and silver so far as possible the same. From his results the author concludes that the effect of the sulphides is not uniform, but depends on the nature of the ore to an extent which demands further investigation. Nevertheless, in assaying pure silver and lead, increasing the proportion of silica increases the loss of silver. In presence of ferrous sulphide large quantities of silica carry silver into the slags; but if only a little silica is present, the iron sulphide reduces the loss. It is therefore impossible to insure obtaining the same yield of silver from different ore mixtures, even if the amount of metal in the assay is actually the same. F. H. L.

Estimation of Gold and Silver in Pyrites. W. Buddëus. (*Chem. Zeit.*, 1900, xxiv., 922.)—The ore is reduced till it will pass through a sieve of 300 or 400 meshes per square centimetre—extreme fineness is not essential—and 100 grammes are taken for analysis. If, however, it contains less than 50 grammes of gold per 1,000 kilos., 200 grammes should be employed, all the following figures being then doubled correspondingly. It is placed in a 70 c.c. biscuit crucible, lightly covered, and put into a bright red-hot muffle for thirty or forty-five minutes until the sulphur flame disappears. The crucible is then cooled, and the coherent mass is thrown out into a beaker or basin, wiping the crucible with a brush. Without breaking up the lump, it is covered with 250 c.c. of 1 : 1 hydrochloric acid, free from arsenic. After standing one hour in a warm place, a second 250 c.c. of acid are added, and the whole is boiled. The liquid is diluted to 1 litre, allowed to settle, filtered, and the precipitate, which contains all the gold, silver, silica, and alumina of the ore, is washed two or three times with water. The paper and contents are dried, introduced into a 150 c.c. Hessian crucible with 50 grammes of lead and 5 grammes of borax, and well mixed. If preferred, 50 grammes each of lead acetate and dry sodium carbonate may be used instead of the metallic lead. The whole is melted; the button is taken out, cupelled, and finally parted in the ordinary way. The preliminary removal of the iron sulphide prevents loss of gold and silver in the large mass of slag otherwise obtained, while it avoids the very high temperatures needed to convert the sulphide into a silicate, which tends to cause volatilization. For these and other apparent reasons the process is much cheaper to work; it gives considerably higher figures than the usual method—figures which agree much better among themselves, and it is therefore more accurate and trustworthy. F. H. L.

Estimation of Mercury in Urine. J. Malkes. (*Chem. Zeit.*, 1900, xxiv., 816.)—This process, devised by Stukawenkow, has been in use in Russia for several years, but appears to have been overlooked elsewhere. It depends on the precipitation of the mercury with albumin and the final production of mercuric iodide. Five hundred c.c. of urine in a 1-litre flask are mixed with 5 c.c. of egg albumin and

15 or 20 drops of acetic acid, then placed on a water-bath and heated for fifteen to twenty minutes. The whole is poured into a beaker, and allowed to settle for twenty minutes; the clear liquid is run away, and the deposit collected on a filter. The paper and its contents are laid on a porous tile for a few minutes. The precipitate is then removed from the paper, brought into a small cylinder, and covered with 50 c.c. of strong hydrochloric acid, a spiral of copper wire being immersed in the liquid. In about fourteen or sixteen hours all the mercury has amalgamated with the copper, and the acid has become dark in colour. The wire is washed with water, alcohol and ether, and dried between filter-paper. It is next dropped into a tube 5 millimetres in diameter with a crystal of iodine, and heated cautiously till the mercuric iodide forms a red sublimate on the walls of the tube. The amount of mercury in the original urine is estimated by comparing the size of this ring with that yielded by a urine to which a certain quantity of mercuric chloride has been added, it being convenient to prepare a series of such tubes to act as standards. The method is very exact, especially when only small amounts of mercury are present.

F. H. L.

Estimation of Tungsten in Ores. F. Bullnheimer. (*Chem. Zeit.*, 1900, xxiv., 870.)—Although the determination of tungsten in high grade or purified ores presents no particular difficulty, the presence of inferior material, and especially of phosphorus and arsenic compounds, complicates the process. One or two grammes of the finely-powdered mineral are best mixed with 4 grammes of sodium peroxide and 3 grammes of sodium hydroxide in a nickel crucible, and heated over a small flame till the mass softens. The temperature is then raised till the bottom of the crucible begins to glow and the mixture is thoroughly fluid, stirring continuously. After the melt has solidified, but while it is still hot, crucible and contents are thrown into water. If manganese causes the liquid to be green, it is decolorized with hydrogen peroxide, then cooled, diluted to 250 c.c. and filtered. To half the filtrate 20 grammes of ammonium nitrate are added, and after the tin and silica have settled, a sufficient quantity of magnesium nitrate solution is run in gradually and with agitation. After 6 or 12 hours the precipitate is filtered off and washed with ammonia and water; the filtrate is rendered slightly acid with nitric acid, cooled if necessary, and treated with 20 or 30 c.c. of a solution of mercurous nitrate (200 grammes warmed with 20 c.c. of strong nitric acid and some water, diluted to 1 litre and kept over mercury). After standing a few hours, ammonia is introduced till the liquid is only faintly acid, and the whole is allowed to rest until the supernatant solution is clear. The precipitate of mercurous tungstate is collected, washed with water containing mercurous nitrate, dried, and ignited first over a Bunsen and then over the blowpipe to constant weight in an uncovered crucible.

The sodium hydroxide in the fusion makes the melt more liquid and preserves the crucible. The nitrates of ammonium and magnesium must be employed, as their chlorides and sulphates would interfere with the mercurous reagent. It is very important not to add the magnesium nitrate till the ammonium nitrate has thrown down the tin and silica. If a considerable amount of molybdenum occurs in the sample, which is not often the case, much time is required to volatilize it completely

out of the final tungsten precipitate. The process may then be hastened by igniting shortly, adding ammonium chloride, and igniting again, first in the covered, afterwards in the uncovered crucible. F. H. L.

The Determination of Tungsten in Ores and Residues. H. Borntträger. (*Zeit. anal. Chem.*, 1900, xxxix., 361, 362.)—One gramme of the ore is fused with 10 grammes of sodium hydroxide for an hour, when the clear mass is extracted with hot water, the whole made up to 250 c.c., cooled and filtered. One hundred c.c. of the filtrate are added to a mixture of 15 c.c. of concentrated nitric acid, and 45 c.c. of concentrated hydrochloric acid, and evaporated to complete dryness in a porcelain dish. The residue is taken up with a solution containing 10 per cent. of ammonium chloride and 10 per cent. of concentrated hydrochloric acid, filtered, and the residue, which contains silica and tin oxide in addition to tungstic acid, dissolved in warm ammonium hydroxide, with which the filter is also washed. The solution is run into a mixture of 15 c.c. of nitric acid and 45 c.c. of hydrochloric acid, and the whole again evaporated to dryness. The tungstic acid, as thus obtained, is free from silica and tin oxide.

In the analysis of metallic tungsten, which also contains oxygen, carbon, antimony, iron, and silica, a preliminary ignition for thirty minutes is necessary, and in the fusion an addition of a little nitre to the sodium hydroxide is advisable.

Metallic tungsten, as met with in commerce, usually contains from 95 to 97·5 per cent. of tungsten.

The method of precipitation with mercuric nitrate is stated to give very uncertain results, owing to the readiness with which the finely divided mercuric tungstate passes through the filter. The method described above is now used in all the larger metallurgical works in Germany. C. A. M.

A Method for the Rapid Estimation of Lime. W. H. Hess. (*Journ. Amer. Chem. Soc.*, 1900, xxii., 477.)—The lime is precipitated and separated as oxalate in the usual way. The filter-paper is burnt. Then the lime in the crucible is mixed with an equal bulk of ammonium nitrate and about twice as much ammonium sulphate. The crucible is then covered with a lid, and ignited cautiously until no more fumes are observed. Intense ignition is unnecessary, and is to be avoided. The lime is thus converted into sulphate, and is weighed as such. The results quoted are all 0·1 per cent. too low. A. M.

The Examination of Commercial Thorium Nitrate and Incandescence Mantles. W. Muthmann and E. Bauer. (*Berichte*, xxxiii., 2028-2031.)—It is well known that the thorium preparations obtained from monazite sand show enormous variations in their capacity for emitting light, so much so that the German factories have their mantles officially tested at Charlottenburg and Karlsruhe, and sell them with a guarantee as to their emission power.

This difference is altogether apart from the prejudicial influence of common impurities such as phosphoric acid, alkalies, and iron, chromium, and aluminium oxides.

The authors have examined different samples of commercial thorium nitrate by their cathode-luminescence method (*Berichte*, xxxiii., 1748 and 1760) and have found them to invariably contain impurities in the form of rare earths. In this method it was found best to convert the nitrates into anhydrous sulphates, and to subject these to the influence of cathode rays in an exhausted tube, the phosphorescent light emitted being decomposed by a prism, as used by Crookes. In all the preparations examined, the occurrence of lines of gadolinium and yttrium was proved, and it was established that the former greatly preponderated.

One sample of commercial nitrate (840 grammes) was dissolved in 5 litres of water and fractionally precipitated by means of a solution of potassium chromate, steam being introduced under a pressure of three atmospheres during the precipitation (ANALYST, xxv., 130).

The first six fractions consisted of thorium chromate yielding a pure white oxide (320 grammes). On further fractionation, a slight quantity of a dirty olive-green chromate was precipitated, and the residual solution, from which nothing more was precipitated by potassium chromate, yielded on the addition of potassium hydroxide precipitates of the hydroxides of gadolinium and yttrium. These gave about 0·5 gramme of the oxides, which, fromt heir luminescence spectrum, were found to consist mainly of Gd_2O_3. The mixed oxides from the olive-green chromate, which was the last chromium precipitate obtained, were found by spectrum analysis to consist principally of neodymium and praseodymium oxides.

Hence the authors concluded that the total amount of these impurities was at least 0·3 per cent.

The pure thorium nitrate thus obtained was mixed with the requisite amount of cerium ammonium nitrate, and photometric tests made with mantles prepared from the mixture. It was found that the addition of the above impurities has a marked injurious influence on the emission-capacity of the mantle, but that the ordinary impurities in the cerium oxide (neodymium and lanthanum salts, etc.) compose such a trifling proportion of the total earths in the mantle that they appear unlikely to have any appreciable effect upon the light. C. A. M.

The Estimation of Cerium in Cerous Salts. G. v. Knorre. (*Berichte*, 1900, xxxiii., 1924-1929.)—In order to use the author's volumetric method (ANALYST, xxiii., 191) with cerous salts it is necessary to convert these quantitatively into ceric salts. This may be readily effected by adding sulphuric acid to the solution, then ammonium persulphate in excess, and heating the liquid to the boiling-point. Even a very dilute solution becomes yellow in a few minutes when thus treated, so that the reaction appears to afford a sensitive test for cerium. An essential condition is that the sulphuric acid must not be added in too great an excess, since otherwise the hydrogen peroxide liberated would reduce ceric salts when formed.

The quantitative oxidation is carried out as follows: The solution of the cerous salt is acidified with the smallest possible quantity of sulphuric acid, and, after the addition of ammonium persulphate, boiled for one or two minutes. The beaker is cooled in water at about 40° to 60° C., a second portion of persulphate introduced,

and the liquid again heated to the boiling-point. After one more cooling, a third portion of persulphate is added, and the liquid now boiled for ten to fifteen minutes, after which it is advisable to add some more sulphuric acid to destroy the excess of persulphate as far as possible. From 0·2 to 0·3 gramme of cerium require for oxidation about 3 grammes of ammonium persulphate. This is dissolved in water, and about half added for the first boiling, and one-fourth for the second and third boilings.

When the oxidized solution is perfectly cold, the cerium is titrated with standardized hydrogen peroxide until colourless, and the excess of the latter titrated back with standard permanganate. C. A. M.

The Determination of Potassium in Potassium Salts. H. Neubauer. (*Zeit. anal. Chem.*, 1900, xxix., 481-502.)—For the estimation of potassium in various mixtures of sulphates and chlorides of potassium, sodium, calcium and magnesium, which are commonly known as "potash salts," the author recommends the following method, each stage of which is elucidated by experiments described in detail in the paper:

Twenty-five c.c. (=0·5 gramme of substance) of an aqueous solution of the salts are evaporated together with a few drops of hydrochloric acid and sufficient platinum chloride to leave a slight excess after the formation of the double chloride.

The evaporation is continued to dryness, but the heating must not be continued longer than is necessary.

After cooling the mass is moistened with about 1 c.c. of water, and carefully rubbed with the end of a flattened glass rod. It is next treated with at least 30 c.c. of commercial alcohol (93 to 96 per cent.) in successive portions of 10 c.c., and thoroughly rubbed with the glass rod after each addition. If much sodium or magnesium sulphate be present, the salt is first soft and pasty, but eventually becomes hard and crystalline.

The basin is now covered and allowed to stand for thirty minutes, the precipitate receiving an occasional rub during the time. It is then filtered into a Gooch crucible containing asbestos, after being washed with alcohol in the dish, as far as possible. On the filter it is washed with ether, and the latter removed by drawing a current of air through the filter.

The crucible is next gently heated while a current of hydrogen or coal-gas is introduced through a hole in the lid. The flame must be very small at first, in order to prevent decrepitation of the crystals. After five minutes it may be slightly increased, so that the bottom of the crucible is at a dull red heat in the middle. The reduction is continued for twenty minutes. The contents of the crucible are first moistened with cold water, and then washed with hot water, with the aid of the filter-pump, about fifteen times, and then with dilute nitric acid (5 per cent.) for about thirty minutes without the use of the pump, the acid in the crucible being added to as required. Finally, the acid is drawn off and the residual platinum washed with hot water, ignited and weighed.

The weight multiplied by 0·48108 gives the corresponding amount of potassium

oxide (K_2O). It is advisable to repeat the washing with nitric acid to insure the complete removal of the salts.

As there is a chance of error through the asbestos losing in weight during the washings, the author states that he has made successful experiments in the direction of using spongy platinum instead of asbestos in the Gooch crucible. C. A. M.

Diphenylcarbazide as a Reagent for Certain Metallic Compounds. A. Caseneuve. (*Journ. Pharm. Chim.*, 1900, xii., 150-156.)—The formation of the diphenylcarbazones of copper and mercury can be utilized for the detection of traces of those metals. Salts of silver, gold, and zinc (in concentrated solution) have also an action on the carbazide, but the faint rose colour produced cannot be confused with those of copper (violet) and mercury (blue). Certain oxidizing agents—*e.g.*, ferric salts and chromates—also produce characteristically coloured compounds.

Detection of Copper.—The diphenylcarbazide should be purified by crystallization from glacial acetic acid, and drying at 80° C. For the detection of copper in solutions containing 1 part or more in 1,000, a few drops of a recently prepared 1 per cent. alcoholic solution are added to the previously neutralized liquid, when an intense violet colour is immediately developed. In solutions of 1 : 10,000 or more, 5 c.c. of a cold solution of the reagent in benzene are shaken with 10 c.c. of the neutral solution under examination. Solutions of 1 : 100,000, which give no coloration with potassium ferrocyanide, show the author's reaction distinctly.

Detection of Mercury.—The test is made in the same way as for copper. A deep-blue tint, which is still visible in a dilution of 1 : 100,000, is obtained. It can be distinguished from the copper coloration by the fact that in a solution of a mercurial salt containing 1 : 10,000 it does not disappear on the addition of a drop of concentrated nitric acid.

Detection of Iron in Ferric Salts.—These give a red coloration with the benzene solution of the diphenylcarbazide, which is perceptible in solutions containing 1 : 100,000. On adding a drop of glacial acetic acid or of a 1 per cent. solution of potassium ferrocyanide the colour nearly disappears, the benzene only retaining a faint brown shade. The reaction is not so sensitive as those given by copper and mercury.

Detection of Chromates.—The solution of the chromate is rendered strongly acid with acetic or hydrochloric acid and a little powdered diphenylcarbazide introduced. An intense violet coloration is developed on shaking the liquid, and this reaction is sensitive for solutions containing only 1 part of bichromate in 1,000,000. The colour can be distinguished from that given by copper or iron by the fact that it is not extracted from its aqueous solution by benzene; moreover, iron and copper do not give a violet coloration in the presence of an excess of free hydrochloric acid. A chromate can thus be detected in the presence of a ferric salt. C. A. M.

The Use of Sodium Thiosulphate in Quantitative Analysis. F. Faktor (*Zeit. anal. Chem.*, 1900, xxxix., 345-354.)—The author has made numerous experiments on the behaviour of sodium thiosulphate with different metallic salts, with the

object of determining to what extent it could replace hydrogen sulphide in quantitative analysis.

Chromates.—On boiling together aqueous solution of potassium bichromate and sodium thiosulphate, a brown precipitate of hydrated chromic chromate is obtained, whilst part of the bichromate is converted into normal chromate. After continued boiling with thiosulphate in excess, the whole of the chromate is precipitated, and the precipitate can be ignited and weighed as chromic oxide.

Potassium chromate when treated in the same way does not yield a precipitate of chromic chromate, which, however, is obtained quantitatively on adding ammonium or magnesium chloride to the liquid before boiling. Sodium chloride, however, does not induce a precipitate.

Chromic salts are decomposed on continued boiling with sodium thiosulphate, yielding chromic hydroxide as in the equation:

$$Cr_2Cl_6 + 3Na_2S_2O_3 + 3H_2O = Cr_2O_6H_6 + 6NaCl + 3SO_2 + S_3.$$

As this method did not yield altogether satisfactory results, the precipitation was rendered more complete and rapid by adding to the solution of the chromic chloride a definite quantity of potassium chromate solution of known strength, together with some ammonium chloride, boiling, and deducting from the chromic oxide, obtained on igniting the precipitate, the amount due to the chromate added.

Lead Salts.—Good results are given by the following method: The hot solution of lead nitrate is mixed with ammonium chloride, and the resulting precipitate of lead chloride allowed to dissolve in the hot liquid. On now adding a hot concentrated solution of sodium thiosulphate, the lead is precipitated quantitatively as sulphide.

Mercuric Salts.—On treating a hot solution of mercuric chloride with sodium thiosulphate, the mercury is quantitatively precipitated as sulphide, and the precipitation is much more rapid than with hydrogen sulphide.

Silver Salts.—These react at the ordinary temperature with sodium thiosulphate, yielding a white precipitate ($Ag_2S_2O_3$), which on warming becomes yellow and finally black (Ag_2S). The tabulated results given by the author show that the method is suitable for the quantitative estimation of silver.

Nickel and Cobalt Salts.—Nickel salts give a black precipitate of nickel sulphide on being boiled with sodium thiosulphate, but too slowly and incompletely for a quantitative estimation, even after the addition of ammonium chloride. The same difficulty was experienced in the case of cobalt salts. C. A. M.

The Reduction of Nitrates by Lactic Acid. L. Vanino and O. Hauser. (*Zeit. anal. Chem.*, 1900, xxix., 506, 507.)—When bismuth nitrate is heated with lactic acid on the water-bath, it begins to decompose, and at a higher temperature (200° to 210° C.), metallic bismuth separates. In a quantitative experiment the authors obtained from 10 grammes of the salt about 4 grammes of black bismuth powder, or about 93 per cent. of the calculated quantity. Basic bismuth nitrate required a higher temperature for its reduction.

Mercuric nitrate is slowly decomposed above 100° C. with the deposition of mercury, and a similar reaction takes place in the case of mercurous nitrate, though more slowly.

Cadmium, silver, and lead nitrates all yield mirrors of their respective metals.

Cobalt nitrate heated with lactic acid at 360° to 370° C. yields a black magnetic powder, consisting of a mixture of carbon and metallic cobalt. The yield of the latter was 1·6 grammes as against 1·9 grammes calculated, and it constituted 52·6 per cent. of the powder. Nickel nitrate is similarly reduced. A strongly magnetic powder was obtained in the same way from ferric nitrate, but this was not proved to contain metallic iron.

With the chlorides and sulphates of these metals there was either no reduction or only a very slight one. C. A. M.

Estimation of Chlorides, Chlorates, and Perchlorates when mixed together. N. Blattner and J. Brasseur. (*Chem. Zeit.*, 1900, xxiv., 793.)—The author's process is based on the fact that sulphurous acid reduces chlorates to chlorides, but is without influence upon perchlorates. Twenty or 40 grammes of the sample are dissolved in 200 c.c. of water; in 50 c.c. of the solution the chlorine of the chlorides is determined by titration with silver. Another 50 c.c. are saturated with a current of pure sulphurous acid gas, or mixed with 50 c.c. of a saturated aqueous solution of the gas, then boiled gently, treated with precipitated chalk to destroy the free sulphuric acid, cooled, and titrated as before, giving the chlorine of the chlorides and chlorates together. The amount of perchlorate is deduced by carrying out the process already described by the authors (ANALYST, 1899, xxiv., 26). Examples are quoted showing that sulphurous acid has no effect upon perchlorates. The authors remark that they have analysed a large number of samples of "refined" Chili saltpetre, and have found between 0·15 and 1·0 per cent. of $KClO_4$, but they have never yet detected the presence of chlorates. F. H. L.

Erdmann's Reagent for Nitrites in Water. H. Mennicke. (*Zeits. angew. Chem.*, 1900, 711.)—This article describes further minute investigations into the value and delicacy of amido-naphthol-disulphonic acid as a test for nitrites, or as a means of estimating them in water. The results simply confirm the statements already made by Mennicke (this vol., p. 167) and by Erdmann (p. 81) as to the superiority of the new reagent over starch, Riegler's test, and all the various modifications of the Griess α-naphthylamine method. Erdmann now employs paramidobenzoic acid instead of the original aniline-*p*-sulphonic acid, a change which halves the time (now five minutes) required for diazotation, while, as before, the colour attains its maximum in one hour. Fluorescence is very much less noticeable than with any other of the tests; in fact, it can only be seen by examining the upper surface of the liquid with oblique light against a black background. In very extreme dilutions the red colour due to nitrites may be so faint as to be capable of confusion with the fluorescence colour; while by standing for a long time in an open tube the mixture of water and reagent may acquire a mauve or heliotrope tint. To eliminate the

former source of error, a stoppered tube charged with the reagent and doubly-distilled water (distilled over chalk and permanganate) should be kept for comparison; whereas the latter will be avoided by working always in closed tubes. With these precautions the utmost limit of delicacy becomes 1 part of $NaNO_2$ per 2,000,000,000. It is worth noticing that in sewage the Erdmann colour is more of a heliotrope than the wine or Bordeaux-red produced by pure solutions of a nitrite; but if a few drops of strong hydrochloric acid are added to the water the colour becomes normal. The colouring matter always remains in solution, and is therefore susceptible of measurement, even if the liquid contain as much as 0·025 gramme of $NaNO_2$ per litre. Various tables are given. F. H. L.

Simple and Accurate Method for estimating Dissolved Oxygen in Fresh Water, Sea Water, Sewage Effluents, etc. Letts and Blake. (*Chem. News*, vol. lxxxii., pp. 163, 164.)—The authors employ an ordinary separating funnel (332·5 c.c.), which is filled with the water under examination. Seven c.c. of the water are then drawn off and replaced by 5 c.c. of standard ferrous sulphate solution (48 grammes per litre), followed by 2 c.c. of strong ammonia. The stopper being inserted in the funnel, without introducing any air-bubbles, the contents are mixed by gentle agitation, and the resulting greenish-turbid mixture becomes darker in colour as the oxygen absorption progresses. After fifteen minutes the funnel is inverted and about 8 c.c. of dilute (50 per cent.) sulphuric acid are run in through the tube in order to dissolve the iron hydrates; the clear solution is then transferred to a porcelain dish and titrated with permanganate or bichromate (the latter giving better results with sea water and sewage effluent), preferably of such strength that 1 c.c. = 1 c.c. of dissolved oxygen at 0° C. and 760 mm. C. S.

The Quantitative Estimation of Ozone. O. Brunck. (*Berichte*, 1900, xxxiii., 1832-1842).—The method most commonly employed for the estimation of ozone consists in titrating the iodine liberated by the gas from a neutral or acid solution of potassium iodide. As many have failed to obtain reliable results by this method, the author has made experiments to determine the cause. He finds that when a neutral solution of potassium iodide is used, the quantities of ozone found are from 50 to 60 per cent. lower than with an acid solution.

By using an acid solution of potassium iodide the author states that reliable results are obtained, and that the only source of error is the decomposition of the hydriodic acid if the solution be concentrated; and this may be allowed for by a blank determination.

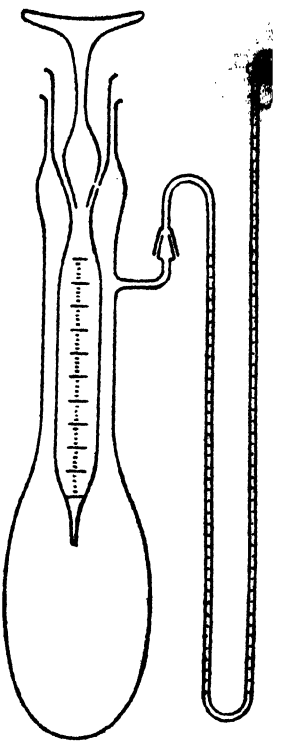
Either sulphuric or acetic acid may be used, but it is advisable to always employ the calculated quantity. In testing ozonizers, the most suitable method is to pass the current of gas through the acidified iodide solution for a given interval of time, or when frequent estimations are required, a proportional side current, as proposed by Rabe, may be employed.

The most suitable strength in most cases for the iodide solution is one-fifth normal, and after the absorption an aliquot portion should be titrated with $\frac{1}{100}$ N. thiosulphate

solution. Nitrous acids are best removed by washing the gas with sulphuric acid, which, unlike the potassium hydroxide solution advocated by Cossa, does not attack the ozone. C. A. M.

APPARATUS.

An Apparatus for measuring Gas under Constant Volume. M. A. Job. (*Bull. Soc. Chim.*, 1900, xxiii., 288-291.)—The apparatus shown in the figure was devised with the object of measuring the increase of pressure in a closed vessel caused by the evolution of a gas in the course of a chemical reaction.



It consists of a glass vessel, the neck of which has a side tube attached to a mercury manometer of narrow gauge. A tube of special construction terminating in a graduated pipette is fitted into the neck of the flask, and this tube is closed by a stopper, which is in the form of a hollow key, with an opening in its side, which can be turned so as to coincide with a corresponding opening in the pipette. When the key is thus turned the liquid in the pipette runs out, being replaced by the air in the flask. The capacity of the apparatus is determined once for all by weighing it empty and filled with water.

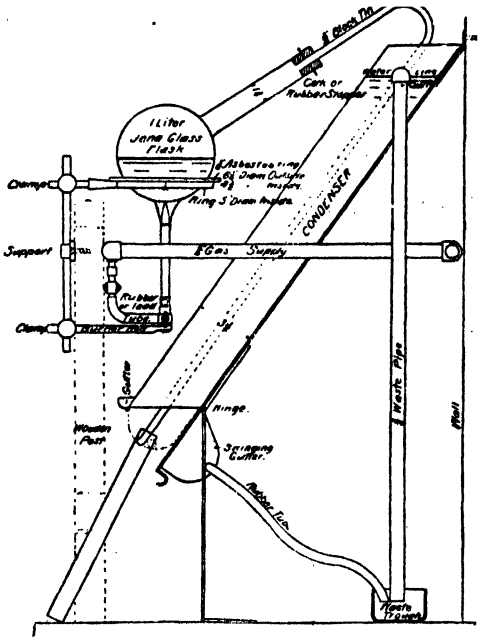
In using the apparatus it should be immersed in a water-bath at the temperature of the room to insure that the initial and final temperatures are the same. The amount of the gas disengaged (reduced to 0° C. and 760 m.m.) is found by the formula $x = \frac{V}{760} \frac{h}{1+\alpha t}$, in which t is the temperature of the water-bath, h the excess of pressure measured, and the last part is to be found in gas-reduction tables. If the apparatus is constructed so as to hold 76 c.c., then $\frac{h}{1+\alpha t}$ expressed in millimetres will give x directly in cubic centimetres. The apparatus is recommended for the determination of carbonates, carbides, sulphides, ammonium salts, and urea.

C. A. M.

Platinum and Brass Electrodes for Analytical Purposes. H. Paweck. (*Chem. Zeit.*, 1900, xxiv., 855.)—This article consists partly of a claim for priority over Winkler in his advocacy of platinum gauze cathodes for electrolytic analysis (ANALYST, 1900, xxv., 55). Paweck, however, prefers a disc-shaped cathode made of wire 0·15 millimetre in diameter, 10 meshes per centimetre, about 30 square centimetres in total area, and with a 1·5 millimetre platinum wire fixed normally to it to serve as a handle and connecting piece. Such a cathode only weighs about 5·5 grammes,

and is therefore cheaper and more convenient than Winkler's gauze cylinder. As anode a perforated disc of platinum foil 6 centimetres in diameter may be employed.

Perfectly good results can be obtained in the deposition of copper, zinc, mercury, etc., by using a cathode of brass wire made exactly as above, a similar electrode constructed of platinum serving as anode. Ten grammes of copper may be deposited by working with a current of 1 to 1·5 ampères at 3 volts at a temperature of 30° C., allowing the action to proceed all night. Copper in white cast-iron can be conveniently determined by treating the original solution with sulphuretted hydrogen, dissolving the precipitate in a little nitric acid, diluting suitably, and electrolyzing with a current 0·2 ampère at 2·2 volts, electrodes 2 centimetres apart. F. H. L.



Apparatus for the Determiantion of Ammonia in Water and of Nitrogen by the Kjeldahl Method. R. S. Weston. (*Journ. Amer. Chem. Soc.*, 1900, xxii., 468.)—The apparatus is so arranged that the distilling-flask and the receiver can both be got at from in front. The condenser-tank is inclined so as to bring the lower end well forward, as shown in figure. Each distillation-flask is supported by a ring, which is clamped to a separate upright rod. A Bunsen burner is supported beneath each flask from the same rod. The upright rods are fixed to a horizontal support. At the bottom of the condenser-tank is a gutter to carry off moisture condensed from the atmosphere. There is also a swinging gutter to carry off the distillate whilst the apparatus is being boiled out ready for a determination. A. M.

An Asbestos Air Bath. O. Böttcher. (*Chem. Zeit.*, 1900, xxiv., 794.)—This piece of apparatus is suggested as a substitute for the water- or sand- bath used in evaporations; it is very simple and cheap, and is very economical in gas. It consists of a plate of asbestos 1·5 millimetre thick, its length and breadth being adjusted according to circumstances. Centrally on the plate is laid a ring of asbestos, 3 or 4 millimetres thick in its walls, 30 to 40 millimetres wide (*i.e.*, high), and of a diameter suitable to the flask or basin to be supported. The apparatus is heated with a Bunsen or an Argand burner, and if it be desirable to maintain the temperature of the bath at one particular point, a small hole may be punched in the ring, through which a thermometer is inserted. F. H. L.

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